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**School of Earth Sciences and Engineering**

**BENEFICIATION OF LOW-GRADE IRON ORE USING NON-  
COKING COAL AS A REDUCTANT**

Full Thesis, Submitted in Partial Fulfilment of the Requirements for

The Award of the Degree of Master of Engineering

In

MATERIALS AND METALLURGICAL ENGINEERING

In the

SCHOOL OF EARTH SCIENCES AND ENGINEERING

Of the

BOTSWANA INTERNATIONAL UNIVERSITY OF SCIENCE AND TECHNOLOGY

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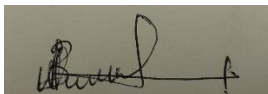
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November 2024

## DECLARATION

I hereby declare that his thesis which, I hereby submit as partial fulfilment of the qualification of: MASTER OF ENGINEERING IN MATERIALS AND METALLURGICAL ENGINEERING, is entirely the result of my own original research, and all findings were conducted at Botswana International University of Science and Technology (BIUST). I affirm that this dissertation has not been submitted to any other academic institution or university for degree certification or a Master of Engineering (MEng).

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## PUBLICATIONS

1. **Bulayani, M.M.**; Raghupatruni, P.; Mamvura, T.; Danha, G. Exploring Low-Grade Iron Ore Beneficiation Techniques: A Comprehensive Review. Minerals 2024, 14, 796. <https://doi.org/10.3390/min14080796>
2. **Bulayani, M.M.**; Raghupatruni, P.; Danha, G.; Mamvura, T. Beneficiation of low-grade iron ore through reduction roasting coupled with magnetic separation. Minerals Engineering 2024, (**to be submitted**)

## ABSTRACT

This research explores the beneficiation of low-grade iron ore from Botswana using non-coking coal as a reductant through the process of reduction roasting and subsequent magnetic separation. The increasing global demand for steel, coupled with the depletion of high-grade iron ore deposits, necessitates the exploration of low-grade resources to maintain a stable iron supply. Most iron ore globally is processed through traditional blast furnace methods, which require high-grade ores. However, the exhaustion of these high-grade deposits has driven efforts to upgrade low-grade ores, which present unique challenges due to their higher impurity content and lower iron concentration. This study explores an alternative approach using abundant local resources in Botswana, such as low-grade iron ore and semi-bituminous coal. The iron ore from the Ikongwe Mine presents a significant opportunity for value addition; particularly as high-quality ore deposits are increasingly depleted globally. Given the considerable reserves of semi-bituminous coal in Botswana, this research investigates the feasibility of employing locally sourced reductants to enhance the iron content of locally low-grade ores, making them suitable for industrial applications. The experimental methodology involved mineralogical and chemical characterisation of both the ore and coal samples, followed by reduction roasting in a muffle furnace at varying temperatures (700°C, 800°C, and 900°C), residence times, and coal-to-ore ratios. Taguchi experimental design was employed to optimise process parameters, including roasting time and coal dosage, ensuring efficient conversion of hematite to magnetite, which was subsequently separated using low-intensity magnetic separation. The key findings show a significant improvement in iron content, increasing from 56% to over 68%, with the optimal roasting temperature identified at 800°C, and a coal dosage of 0.5%. This indicates the potential of Botswana's non-coking coal to serve as a reductant for iron ore beneficiation, achieving recovery rates and Fe grades comparable to established processes. Thermodynamic and kinetic analyses further validate the feasibility of this approach, with activation energies calculated at 159.1 to 160.6 kJ/mol aligning with industry benchmarks. The reaction mechanisms during reduction roasting are detailed through a gas-solid reaction model, highlighting the crucial role of carbon monoxide as a reducing agent and the

importance of maintaining optimal temperature and coal ratios for efficient hematite to magnetite conversion. This study concludes that the integration of locally sourced non-coking coal for iron ore beneficiation is not only technically viable but also holds promise for economic diversification in Botswana, reducing the nation's dependency on iron and steel imports.

## **ACKNOWLEDGMENTS**

I would like to express my profound gratitude to Botswana International University of Science and Technology (BIUST) for providing me the opportunity to conduct my process mineralogy dissertation at the institution. I extend my thanks to my supervisors, Dr. Prasad Raghupatruni, Prof. Gwiranai Danha, and Dr. Tirivaviri Mamvura, for their invaluable guidance and support throughout my research.

I am also grateful to the technical staff for their constant assistance in the lab and for sharing their expertise, which greatly facilitated my work.

Finally, I want to acknowledge my family and friends, particularly my mom, Kentse Bulayani, and my grandmother Tambe Bulayani (May Her Soul Rest in Peace), for their unwavering support and encouragement. Your positivity has been a source of inspiration throughout this journey. Thank you all.

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## ABBREVIATIONS

| Acronym                        | Expansion                                       |
|--------------------------------|-------------------------------------------------|
| AAS                            | Atomic Absorption Spectroscopy                  |
| AG                             | Autogenous (Mills)                              |
| BHQ                            | Banded Hematite Quartzite                       |
| CO                             | Carbon monoxide                                 |
| DOE                            | Design of Experiments                           |
| EDS                            | Energy Dispersive Spectroscopy                  |
| Fe                             | Iron                                            |
| HIMS                           | High intensity Magnetic Separator               |
| ICP-AES<br>Spectroscopy        | Inductively Coupled Plasma Atomic Emission      |
| LIMS                           | Low Intensity Magnetic Separator                |
| MGS                            | Multi-Gravity Separator                         |
| PSD                            | Particle Size Distribution                      |
| QEMSCAN<br>Electron Microscopy | Quantitative Evaluation of Minerals by Scanning |

|       |                                       |
|-------|---------------------------------------|
| ROM   | Run-of-Mine                           |
| SAG   | Semi-Autogenous (Mills)               |
| SEM   | Scanning Electron Microscopy          |
| SR    | Stripping Ratio                       |
| TGA   | Thermogravimetric Analysis            |
| WHIMS | Wet High-Intensity Magnetic Separator |
| XRD   | X-ray Diffraction                     |
| XRF   | X-ray Fluorescence                    |

# CHAPTER 1

## 1.1 Introduction

Iron ore is the primary raw material for producing iron and steel, other ferrous alloys. Iron and Steel are critically important engineering materials worldwide due to its exceptional blend of strength, malleability, and diverse applications. Steel has become essential in our lives with its application across various industries including construction, automotive, infrastructure and manufacturing, and greatly impacts our economic growth. With a lot of countries developing the consumption and demand of iron and steel is growing every day. This high demand of steel and other iron alloys has led to the exhaustion of high-quality iron ore deposits and validates the need for exploitation of alternative steelmaking routes to the conventional route where high grades are used as direct feed. The vast majority of global iron production, over 90%, takes place via the conventional blast furnace process. In this method, coke (a coal-derived fuel) is combined with iron ore to generate molten iron. Iron is the most affordable and widely utilized metal globally. As an element, it is found in a variety of host ores and rock formations (Crowson, 2011; Kim *et al.*, 2022).

Iron(Fe) is a highly abundant element, making up between 2-3% of the composition of sedimentary rocks and almost 8.5% in basalt and gabbro rock types (Wehleeekema *et al.*, 2018). Iron does not occur naturally in its pure, elemental form. Despite its widespread abundance, iron has a relatively low economic value. Consequently, for an iron ore deposit to be deemed commercially viable, it needs to have a high percentage of iron content. The commercially viable grade of iron ore currently ranges between 44 to 60 wt. % Fe. However, a limited quantity of iron ore deposits can be categorized as high-grade (>62 wt. % Fe). Iron ore deposits can be categorized as either low-grade (<65% Fe) or high-grade (65-77% Fe). The difference in iron ore grades is due to their nature chemical composition and greatly influenced by their geological occurrence. These composition

indifference influences iron ore processing routes depending on their characteristics and properties.

### **Iron ore beneficiation significance.**

- **Iron content upgrade:** Iron ore beneficiation is crucial for enhancing the iron content of ore deposits. Significant iron ores exhibit low iron content, making them unsuitable for direct utilization in iron and steel production. Through beneficiation processes impurities are eliminated and the iron content is boosted.
- **Resource Conservation:** Iron ore beneficiation promotes resource utilization by upgrading low/lean grade iron ores, minimizing the need for expansive mining efforts, and preserving valuable iron resources.
- **Economic viability:** Low grade iron ores are deemed uneconomical owing to their large share of impurities and by improving their quality through beneficiation ultimately their value increases making them economically viable for extraction and processing. This helps in maximizing the economic returns from iron ore reserves, which contributes to the sustainability of the mining industry.
- **Environmental Impact Reduction:** Iron ore beneficiation reduces the need for extensive mining activities, minimizing ecosystems disturbance and energy consumption. Upgrading low grade ores eliminates impurities such as phosphorus and sulphur, resulting in reduced environmental pollution during iron and steel production.
- **Global Iron Supply:** Iron ore beneficiation ensures a stable and sustainable global iron supply. As demand for iron and steel grows, beneficiation processes utilize diverse resources, including lower-grade ores and untapped deposits, meeting global demand, and ensuring steady supply.

## **1.2 Background**

Botswana's iron and steel imports in 2021 were valued at approximately \$116.9 million U.S. dollars, which represented a significant increase compared to the prior year when imports of these products were roughly \$60.5 million U.S. dollars. Furthermore, South Africa accounted for nearly

94% of Botswana's imports of articles made from iron and steel (Cowling, 2024). This growing import bill underscores the need for Botswana to develop its domestic iron ore resources to reduce dependence on imports and promote self-sufficiency. The project, focusing on upgrading low-grade iron ore deposits through reduction roasting and magnetic separation, presents a crucial opportunity to tap into the country's unutilized iron ore reserves. By doing so, Botswana could potentially decrease its iron and steel import costs, diversify local industry, and improve economic resilience.

Extensive research(Dwari, Rao and Reddy, 2014; Rachappa, Prakash, and Amit, 2015; Dash, Rath and Angadi, 2019) in recent years has focused on developing methods to exploit and utilize low/lean grade iron ore reserves, due to desirable high-quality iron ore supplies depletion, increasing global steel demand, and the implementation of stricter environmental regulations. High-grade iron ore contains iron content in the range of 66-77%, with marginal impurities such as silica, alumina, and phosphorus. High-grade iron ore processing involves crushing and screening to generate a lump ore product for direct use in iron production. Ores with low Fe in % wt. and high alumina and silica ratios offer poor productivity when treated through a blast furnace for metal recovery(Ghosh and Chatterjee, 2010). Harmful components (including phosphorus and sulphur) present in iron ore must be removed as they degrade the iron and steel quality. Beneficiation of low/lean grade iron ore is aimed at improving the ore chemical properties and mineral content to make suitable concentrates for smelting and reduction processes. The metallurgical processes involved in iron ores beneficiation are different for each ore type and depend largely on mineralogical characteristics of a particular iron ore(Ghosh and Chatterjee, 2010). The associations of the iron values with the gangue minerals, the nature and amount of the gangue in the ore serve an equally crucial role in the selection of beneficiation processes(Abro *et al.*, 2008; Kiptarus *et al.*, 2015). High-quality iron ore satisfies the specifications needed for ironmaking in the blast furnace process.

As of 2024, Botswana is estimated to have over 200 million metric tonnes of iron ore reserves with substantial iron ore reserves are present in diverse localities countrywide including Shoshong, Kopje, Shakawe, and Matsitama(Steenkamp, 2024). Fig. 1 provides localities where these reserves

are situated. On the other hand, Botswana has considerable coal deposits which provide a carbon source (reductant) during iron reduction. Botswana has among the largest untapped coal reserves globally, with estimates exceeding 212 billion tonnes. However, a significant portion, around 77%, of these reserves fall under the 'hypothetical' and 'speculative' categories, meaning their viability is yet to be fully confirmed. It is estimated that as much as two-thirds of Africa's total coal resources are located within Botswana (Grynberg, 2012). Given Botswana's abundant semi-bituminous coal reserves, the country has significant potential to diversify its economy by pursuing advanced coal utilization technologies such as the beneficiation of iron ore, and subsequently, boost her economy with iron making and steelmaking.

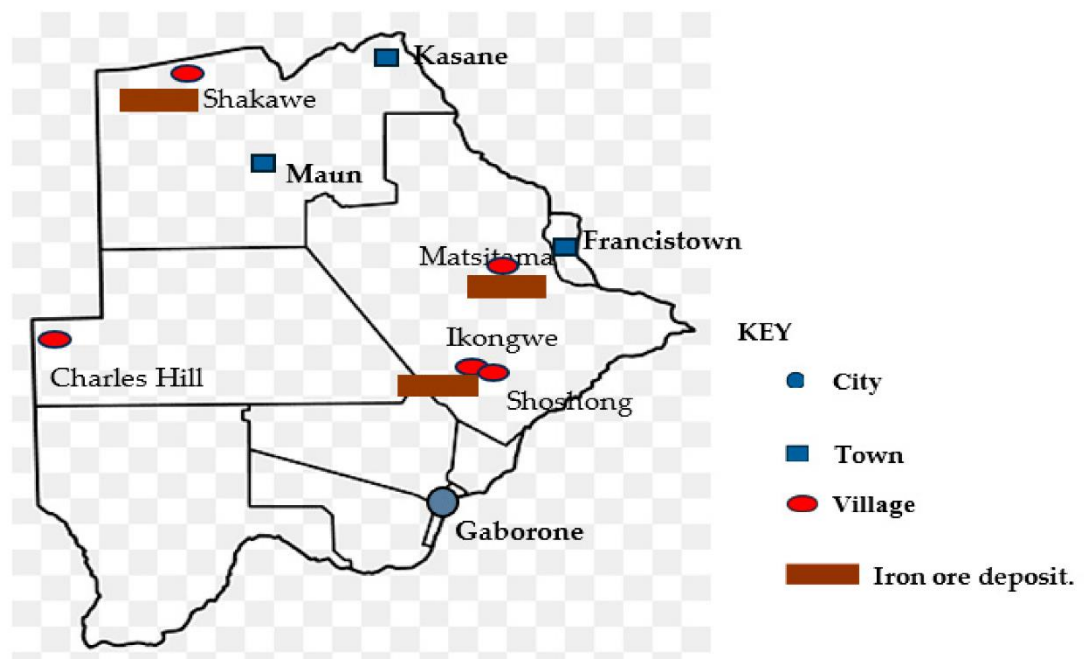


*Figure 1 showing some of the significant iron ore deposit localities.*

### 1.3 Problem statement

- Botswana has significant deposits of low-grade iron ore, which are largely unutilized. These low-grade ores typically contain lower concentrations of iron and higher levels of impurities, making them less suitable for direct use in traditional

iron-making processes. However, they require further beneficiation before being subjected to conventional iron and steelmaking processes.

- The country harbours approximately 212 billion tonnes of coal reserves with the majority of this coal only utilized for power generation purposes. However, some of which could be deployed for the pyro-metallurgical beneficiations of iron ore(s),

Therefore, Botswana is home to potential resources (low grade iron ore and non-coking coal) that could be deployed for production of iron and its alloys. Exploitation of these resources through reduction roasting and magnetic separation will result in great economic diversification and decrease dependence on imports.

### **1.3.1** *General objective/Aim*

The main goal of this study is to beneficiate the low-grade iron ore deposits found in Botswana by utilizing non-coking coal as a reduction complex.

### **1.3.2** *Specific objectives*

- i. Characterisation of local raw materials for mineralogy and chemistry.
- ii. Process Optimization of Iron ore beneficiation by Taguchi and Experimental Validation (Reduction roasting).
- iii. Thermodynamic and reaction kinetics modelling and experimental validation of the process.

### **1.3.3** *Research questions*

1. How can low-grade iron ore from Botswana be beneficiated using non-coking coal as a reductant?
2. What are the optimal parameters for the reduction roasting of low-grade iron ore using non-coking coal?
3. What is the impact of reduction roasting and magnetic separation on the mineralogical and chemical properties of iron ore?

4. How do thermodynamic and kinetic models validate the reduction roasting and magnetic separation process for iron ore beneficiation?
5. Can locally sourced resources be potentially used in the diversification of the economy and reduce imports dependence?

In this study, upgrading the concentration of Fe % in a lean grade iron ore involves a combination of reduction roasting and magnetic separation. This study utilizes the available and abundant non-coking coal as a reduction complex in the reduction roasting process. Low-grade iron ore sample was sourced from the Ikongwe in Botswana, subsequently crushed and ground. X-Ray Diffraction (XRD), Scanning Electron Microscopy combined with Energy Dispersive X-Ray Analysis (SEM/EDX) and X-Ray Fluorescence (XRF) were utilized to assess the iron phases present (XRD), surface morphology (SEM/EDX) and elemental composition (XRF) of the as-received iron ore prior to and after the reduction roasting coupled with magnetic separation process. Subsequent to mineralogical and chemical analysis of both unroasted and roasted iron ores, the feasibility and potential industrial applications of iron ore upgradation through reduction roasting followed by magnetic separation was evaluated via a comparative analysis. The Taguchi Design of Experiments (DOE) approach was employed to optimize the roasting parameters, including temperature, residence time, and coal dosage, ensuring a systematic investigation of their effects on iron grade and recovery. The experimental results were further validated against the model predictions, providing a comprehensive evaluation of the process efficiency and feasibility for industrial applications. Furthermore, thermodynamic and kinetic modelling were also conducted to understand the underlying reaction mechanisms and phase transformations during the reduction roasting process.

## **CHAPTER 2**

### **LITERATURE REVIEW**

In iron and steel making industries iron is the principal raw material with hematite and magnetite being the main iron ore resources. Globally, iron ore reserves are estimated at around 190 billion tons, with an iron content of roughly 87 billion tons. The four countries possessing the largest iron ore reserves are Australia, Brazil, Russia, and China.

The main producers and exporters of iron ore in Africa are South Africa, Mauritania, and Algeria. Iron ore deposits, though in smaller amounts, are also present in several other African countries, including Mozambique, Tunisia, Swaziland, Morocco, Egypt, as well as Niger, Tanzania and Nigeria (Mamaila, no date). There are also countries with significant iron ore deposits that have not been commercialized yet as of 2020 including Botswana among others.

#### **2.1 Iron ore deposits**

Iron is a highly abundant element that is widely used across the globe. The iron content in rocks varies significantly, varying from an average of 2-3% in sedimentary rocks to approximately 8.5% in basalt and gabbro (Wehleeckema, 2017). Iron is primarily sourced from iron ores, which are mineral deposits abundantly found across the globe. Mineral deposits arise from processes that serve to concentrate elements into distinct mineral forms or significantly elevated concentrations within other minerals. When the concentration is high enough for profitable extraction from the earth, these deposits are termed ores(Le, no date). For a mineral deposit to be classified as an iron ore, it must possess adequate size, iron concentration, and compositional and physical attributes to enable it to serve as a source of iron, either currently or in the future(Kennedy and Exploration (U.S.), 1990).

Botswana has significant iron ore deposits, initially found in various localities including Nxamasera, Kopje, Ikongwe, Matsitama and Okavango with an estimated total of over 200 million metric tonnes of resources(Steenkamp, 2024). Vision Ridge Directors who are investors in the Ikongwe Iron ore project had this to say “iron ore resource will yield approximately 1 million

metric tons annually over a ten-year period, with an iron concentration of up to 65%, commonly known as 65% Fe” (Ramaphane, 2021). Some of the prominent iron ore reserves and their abundances are provided in Table 1.

*Table 1. Outlines the localities of iron ore deposits found in Botswana and their respective quantities (Brian Benza, 2012; Steenkamp, 2024)*

| <b>Deposit locality</b> | <b>Abundance<br/>(million tonnes)</b> | <b>Fe %</b> |
|-------------------------|---------------------------------------|-------------|
| <b>Kopje</b>            | 60                                    | 60          |
| <b>Mmamabula</b>        | 100                                   | 56          |
| <b>Nxamasera</b>        | 40                                    | 61          |
| <b>Shakawe</b>          | 50                                    | 68          |
| <b>Ikongwe</b>          | 60                                    | 59 - 61     |
| <b>Tsodilo Hills</b>    | 441                                   | 30-42       |
| <b>Matsitama</b>        | 45                                    | 56          |
| Range                   | <b>40-441</b>                         | 30-68       |

## **2.2 Iron ore mining**

Surface and underground mining are the two main methods used in the extraction of iron ores from their deposits. The choice of mining technique is mainly influenced by how close the ore deposit is to the surface of the ground. Most iron ore reserve(s) in the world are found within proximity to the earth surface and as a result most mining operations are surface mining. However, there are some underground mines that are still operational to date (Stace, 2015).

Most surface mining operations use open-pit or open-cut methods for iron ore extraction (US Environmental Protection Agency, 1994). These methods are the least expensive and relatively less sophisticated extraction techniques. The downside is the low-cost effectiveness relative to the equipment used and the substantive stripping of overburden. Surface mining entails stripping away the overburden of soil and rock to reveal the ore body beneath. After exposure, the ore body is

drilled and blasted with explosives for easy transportation to a processing plant for mineral extraction. Overburden stripping ratios are an important economic consideration in surface mining and determine whether a resource will be exploited or not. The stripping ratio is a measure of the unit overburden material stripped from the surface for each unit of ore mined.

$$\text{Stripping ratio (SR)} = \frac{\text{Volume of Overburden Stripped}}{\text{Volume of Ore Mined}}$$

Or, if considering mass instead of volume:

$$\text{Stripping ratio (SR)} = \frac{\text{Mass of Overburden Stripped}}{\text{Mass of Ore Mined}}$$

Underground mining techniques are preferred when the deposit depth or the stripping ratio required of the deposit are considered not economically viable for surface mining operations. Although deep mining is rare in the current era, the Swedish Kiruna mine is an example of a well-established underground iron ore mine which is largely aided by automation technologies and the use of remotely controlled equipment(Stace, 2015).

### **2.3 Characterisation of iron ores**

The iron and steelmaking industries are faced with the challenge of dwindling high-grade iron ore reserves, compelling them to increasingly utilize low-grade ores that necessitate beneficiation prior to ironmaking. Low-grade ores are often associated with complex mineralogy and intricate associations with unwanted gangue minerals that make necessary the application of advanced techniques for mineral extraction. To this end, characterization of iron ores has become a critical component of metallurgical processing of iron ores. Characterization of iron ore is a vital technique employed for the quantitative and qualitative assessment and categorization of iron mineral deposits (Kiptarus *et al.*, 2015). Characterization techniques provide data on the characteristics of mineral deposits, detailing the mineralogical and chemical makeup of an ore. This information is crucial in the development of efficient process flow diagrams for iron extraction as well as

optimization of downstream processes as it allows the user to predict the ore behaviour during processing (Abro *et al.*, 2008; Seifelnassr, Moslim and Abouzeid, 2013).

Mineralogical characterization techniques are used to determine major minerals and gangue minerals associated in an ore, their associations, and liberations (Krishna *et al.*, 2013; Dwari, Rao and Reddy, 2014). XRD and microscopic techniques are fundamental in the mineralogical investigation of iron ores. Image analysis by advanced microscopy is an important method in the identification and quantification of mineral particles. Advanced Scanning Electron Microscope (SEM) or Reflected Light Optical Microscope (RLOM) – based systems integrated with digital image analysis have been developed in recent years (Tonžetić, Duncan and Bramdeo, 2014; Delbem *et al.*, 2015; Tonžetić, 2022). QEMSCAN (EDS – SEM) is an automated energy dispersive spectrometer attached to scanning electron microscopes, capable of performing particle mapping analysis (Delbem *et al.*, 2015). This system combines Back Scattered Electron (BSE) images with mapping analysis to accurately distinguish mineral species in a sample (Ayling *et al.*, 2012). The chemical analysis of iron ore is typically conducted using methods like inductively coupled plasma atomic emission spectroscopy (ICP-AES), X-ray fluorescence (XRF), atomic absorption spectroscopy (AAS) and pycnometer.

**X-ray diffraction (XRD)** - X-ray diffraction (XRD) is a technique used for compositional analysis that helps identify the mineral phases present in an iron ore sample by examining their crystalline characteristics (de Villiers and Lu, 2015). X-ray diffraction (XRD) not only reveals details about the crystal structure and lattice parameters of both crystalline and amorphous phases, but it also offers qualitative insights into the anions and cations present within the crystalline framework of the sample, extending its application beyond mere compositional analysis of ore samples. This information, in turn, enhances comprehension of the chemical reactions occurring within the ore deposits (de Villiers and Lu, 2015). X-ray diffraction (XRD) analysis is performed with an X-ray diffractometer, which directs a monochromatic X-ray beam at the sample while simultaneously varying the angles of the X-ray source and detector in relation to the sample. The resulting diffraction patterns, displayed as peaks of varying intensities at specific angles, are then recorded

and examined. These patterns are unique to the minerals or phases present in the sample, as they rely on the material's crystal size and shape (Bunaciu, Udriștioiu and Aboul-Enein, 2015).

**Scanning electron microscopy (SEM)** – The use of scanning electron microscope (SEM)-based technologies for the quantitative analysis of iron ore is becoming an essential and highly valuable method for characterizing these materials. This is evidenced by the more than 400 automated SEM instruments that have been deployed globally across various research institutions to support this application (Schulz, Sandmann and Gilbricht, 2020). Thorough comprehension understanding of the mineralogical composition of lower grade iron ores, and subsequently characterizing these lower grade ores, will enable more efficient ore processing and enhance the optimization of subsequent smelting operations (Tonžetić, 2022). In scanning electron microscope (SEM) analysis, images are typically captured by detecting back-scattered electrons from the surfaces of the ore. Another advanced technique for mineralogical characterization is Quantitative Evaluation of Minerals by Scanning Electron Microscopy (QEMSCAN) (Poojari, Vardhan and Jathanna, 2023a). QEMSCAN is an automated system that performs image analysis by combining SEM images and EDS (energy-dispersive X-ray spectroscopy) data, enabling modal analysis and assessment of mineral liberation in ore samples (Schulz, Sandmann and Gilbricht, 2020).

**X-ray fluorescence (XRF)** analysis is a non-destructive analytical method employed to identify the elemental composition and concentrations within samples. It enables the rapid identification and quantification of the elements present in solids, liquids, and loose powders without damaging the sample (Saisho and Hashimoto, 1996; Beckhoff et al., 2007). XRF spectrometers have the capability to detect and measure a wide range of elements, from lightweight beryllium up to heavy uranium, and even beyond. They can quantify these elements at trace levels, often ranging from less than 1 part per million (ppm) up to 100% concentration. XRF spectrometers work by measuring the characteristic wavelengths of the fluorescent radiation emitted by a sample when it is irradiated with X-rays (Kamarudin and Ibrahim, 2012). XRF spectrometers identify the elemental composition of a sample by measuring the specific wavelengths of fluorescent X-rays emitted during X-ray irradiation. ISO 9516 is an international standard that outlines the calibration and elemental analysis of iron ore samples using the fusion method. This method aims to identify

a broad spectrum of major, minor, and trace elements in iron ore. Key elements commonly analysed through this approach include iron (Fe), silicon (Si), aluminium (Al), calcium (Ca), magnesium (Mg), sodium (Na), phosphorus (P), potassium (K), titanium (Ti), manganese (Mn), sulphur (S), and chromium (Cr) (Kamarudin and Ibrahim, 2012).

## **2.4 Iron ore grades**

Despite iron being widely abundant in various forms, pure iron deposits of high concentrations are relatively scarce and considered valuable. To be considered economically viable for extraction and processing, an iron ore deposit generally requires a minimum iron content of approximately 25%. In certain situations, this threshold might be reduced if the ore deposit is sufficiently large and can be extracted, concentrated, and transported at a comparatively low cost (Ofoegbu, 2019). Iron ore deposits can vary significantly in their iron content and impurity levels. The higher the iron content and the lower the impurities depict higher iron ore grade. The classification of iron ore grades is based on the weight percentage (wt.%) of iron (Fe) present; high-grade iron ore: Fe content greater than 65 wt.%; medium-grade iron ore: Fe content between 58 wt.% and 64 wt.% and Low-grade iron ore: Fe content less than 58 wt.%(Kiptarus *et al.*, 2015).

Iron is rarely, if ever, found naturally in its pure, elemental form. Iron can be found in various forms on earth, including iron ore, which is a mineral substance that is mined and processed to extract iron. Iron ore is the primary raw material for steel production, an essential material in various industries. As such, iron ore is a highly valuable and important global commodity that is essential to the overall economy. The iron ore content, the presence and levels of contaminants or impurities, as well as other physical characteristics of various iron ore grades allow for differentiation. The following iron ore grades are the most popular.

1. Hematite: The most prevalent and frequently mined iron ore is hematite. Hematite, sometimes spelled haematite, is a dense and hard iron oxide mineral, (ferric oxide;  $\text{Fe}_2\text{O}_3$ ). It is regarded as the most significant iron ore because of its high iron content, approximately 70 %, and its extensive availability in nature (Manning, 1995; Beukes, Gutzmer and Mukhopadhyay, 2003). The term "hematite" comes from the Greek word "haima," which means "blood," reflecting the mineral's distinctive reddish-brown hue.

2. Magnetite: Magnetite is a highly prevalent iron oxide ( $\text{Fe}_3\text{O}_4$ ) mineral that is found in a variety of geological settings, including metamorphic, igneous and sedimentary rocks. Magnetite is regarded as the most extensively mined source of iron, and it also has the highest iron content among iron ores at 72.4%)(Manning, 1995). Magnetite is known for having magnetic characteristics and is frequently utilized in the manufacturing industry(Gupta and Krishnamurthy, 1992).

3. Goethite: Goethite iron ore typically contains between 30% and 50% iron by weight(Schwertmann and Cornell, 2008). Goethite is a common iron-rich mineral which forms through the weathering of other iron-bearing minerals, especially magnetite, pyrite, siderite, and hematite, under oxidising conditions. It is frequently discovered in sedimentary rocks and soils(Fitzpatrick and Schwertmann, 1982).

4. Siderite: also known as chalybite, is an iron carbonate mineral ( $\text{FeCO}_3$ ), that is a common iron ore(Maynard, 2012) which often forms in thin layers alongside shales, clays, or coal seams as sedimentary deposits, and can also occur in metallic hydrothermal veins as a gangue (waste rock) mineral. Siderite contains between 48% and 53% iron by weigh(Fosbøl, Thomsen and Stenby, 2010).

## **2.5 Gangue minerals in iron ores**

Naturally iron ores consist of impurities, collectively referred to as gangue. Gangue consists of the unwanted minerals and materials that are extracted alongside the desired iron ore (Rachappa, Prakash, and Amit, 2015). The presence of elements other than iron in the ore can have both positive and negative impact on the iron ore characteristics, as well as the processing and production of iron and its downstream products like steel. The impurities can impact the properties and behaviour of the iron ore during mining, beneficiation, smelting, and steelmaking (Ghosh and Chatterjee, 2010). To improve the efficiency of the blast furnace, a fluxing agent is typically added to the iron ore feed. Additionally, other chemicals such as reductants are also introduced to enhance the quality of the final iron or steel product.(Fan *et al.*, 2010; Prusti *et al.*, 2021). Ideally, iron ore would consist solely of iron and oxygen. However, iron ore commonly contains a variety of other elements beyond just iron and oxygen. Many of these additional elements present in iron ore are

often considered undesirable or deleterious in the iron and steel industries (Haga, Siame and Shibayama, 2018). Iron ores typically contain four main slag-forming constituents -  $SiO_2$  (acidic oxide silica),  $CaO$  (basic oxides lime) and magnesia (magnesia), as well as  $Al_2O_3$  (neutral alumina). These elements contribute to the formation of slag during iron and steel production. Most iron ores contain an excess of the acidic oxide silica ( $SiO_2$ ) and the neutral oxide alumina ( $Al_2O_3$ ). The ash produced from the coke used as fuel mainly contains these oxides. Consequently, a basic flux like limestone is usually required to be added (Taylor *et al.*, 2001). Silica is a significant element in iron ore that is typically eliminated as slag during smelting. When temperatures exceed  $1300^{\circ}C$ , silica is converted into silicon, which subsequently combines with iron to create an alloy. This silicon content encourages the production of gray iron, which is less brittle and more amenable to finishing compared to white iron, making it the favoured option for casting. Additionally, the presence of silica has been noted to decrease shrinkage and the occurrence of blowholes in castings, resulting in fewer defective products (Strassburger, 1969). Most iron ores have small quantities of alumina, typically found as clays and the mineral magnesium aluminium carbonate hydroxide ( $Mg_6Al_2CO_3(OH)_{16} \cdot 4H_2O$ ). Alumina increases the viscosity of the slag produced during smelting. Increased viscosity results in a slower movement of the furnace charge because the slag thickens, extending the processes taking place inside the furnace. A high alumina content also complicates slag recovery, which may lead to the furnace becoming frozen. Adding a higher proportion of lime flux can help decrease the viscosity of the slag and mitigate these issues caused by elevated alumina levels in the iron ore (Rosenqvist, 2004).

### 2.5.1 Harmful elements

Phosphorus, sulphur, and titanium are some of the common harmful elements present in iron ores. In addition to the more common impurities, iron ores may also contain smaller amounts of less common elements that are considered deleterious. These include copper, vanadium, zinc, nickel, arsenic, chromium, tin and lead.

**Phosphorus** has five primary impacts on the metal; it improves hardness and strength, lowers the solidus temperature, and boosts both fluidity and cold shortness. Whether these consequences are beneficial or not depends on the intended use of the iron (Ghosh and Chatterjee, 2010). The

presence of phosphorus in iron has a strengthening and hardening effect, with increasing concentrations leading to greater strength and hardness. However, when the concentration exceeds 0.2%, iron becomes more susceptible to cold shortness, which refers to brittleness at low temperatures. Phosphorus reduces the liquids temperature, enabling the iron to stay molten for an extended period and enhancing its fluidity. In fact, incorporating just 1% phosphorus can double the distance that molten iron can flow. To manage high phosphorus levels in iron ores, solutions involve choosing ores with reduced phosphorus content or oxidizing the phosphorus during the fining process by introducing iron oxide (Rostoker and Bronson, 1990).

**Sulphur** is a common impurity found in small quantities within many iron ores. During the high-temperature iron smelting process, this sulphur readily dissolves into both the liquid and solid forms of the iron. Even minor amounts of sulphur can have significant and detrimental consequences. The presence of sulphur causes the iron to become "red-short" or "hot-short", meaning it becomes brittle and prone to cracking when heated to high temperatures (Ghosh and Chatterjee, 2010). The degree of this hot shortness is directly correlated with the concentration of sulphur in the iron. As a result, iron ores containing more than 0.03% sulphur content are generally less preferred for use in iron and steel production due to the negative impacts on the final product's mechanical properties and hot workability. Sulphur content in iron ores can be reduced through various techniques, such as roasting, washing, and the addition of manganese. Roasting the ores at high temperatures oxidizes the sulphur, converting it to sulphur dioxide (SO<sub>2</sub>) gas which can then be released or removed by washing. Additionally, adding manganese to iron ore is an effective method for removing sulphur. This occurs because manganese creates a high-melting-point sulphide compound at the high temperatures involved in iron smelting, which are approximately 1610°C. The formation of this stable manganese sulphide prevents the creation of a lower-melting liquid iron sulphide that would otherwise accumulate at the grain boundaries of the iron, leading to the detrimental "hot-shortness" effect (Verhoeven, 2007).

## **2.6 Processing of iron ores**

Iron purity can vary depending on the source and quality of the iron ore used for its production (Ghosh and Chatterjee, 2010). High-grade iron ore contains a higher concentration of

iron and fewer impurities, resulting in a purer final product. Low-grade iron ore, on the other hand, contains a lower concentration of iron and a higher percentage of impurities(Ogbezode *et al.*, 2023). A high grade of iron typically refers to iron that has a high level of purity and a low level of impurities. High-quality, high-grade iron ores typically contain iron concentrations in the range of 65-70%, with less than 3% of impurities such as silica, alumina, and phosphorus(Jankovic, 2022; Ogbezode *et al.*, 2023).

The processing techniques employed for iron ore extraction and beneficiation can vary depending on the quality or grade of the raw ore material. In recent years, there has been an increasing research interest and focus (Dwari, Rao and Reddy, 2014; Rachappa, Prakash, and Amit, 2015; Dash, Rath and Angadi, 2019) on the on the exploration and use of lower-grade iron ore deposits. This has been prompted by several factors, including the gradual exhaustion of high-quality iron ore reserves and the rising global demand for steel products. Additionally, stricter environmental regulations have prompted the need to find ways to process and extract iron from ores with higher levels of impurities in a more sustainable manner. As a result, significant research efforts have been dedicated to developing advanced technologies and techniques to effectively and efficiently process and beneficiate these lower-grade iron ores, allowing them to become a more viable and utilized source of iron for the steel industry.

High grade iron ore typically undergo a relatively simple processing regimen prior to use in iron extraction, with their inherently high iron content, are commonly crushed and screened to produce a lump ore product that can be directly charged into the ironmaking furnaces. Low-grade iron ores with high alumina and silica ratios offer poor productivity in blast furnace ironmaking or direct reduction process for metal recovery(Ghosh and Chatterjee, 2008).

Harmful components present in iron ore must be removed as they degrade the quality of iron and steel. These components include arsenic, copper, chromium, phosphorus, vanadium and sulphur. The concentration of low-quality ores is aimed at improving the chemical and mineralogical composition of the ore to make suitable concentrates for smelting and reduction processes. The metallurgical methods used for the beneficiation of iron ores vary by ore type and are primarily influenced by the mineralogical properties of each specific iron ore (Ghosh and Chatterjee, 2008). The associations of the iron values with the gangue minerals, the nature and amount of the gangue

in the ore are critical factors of consideration in the selection of beneficiation processes used (Abro *et al.*, 2008; Kiptarus *et al.*, 2015).

High grade iron ore meets the quality requirements for blast furnace ironmaking. Beneficiation of high-grade ore involves mining, crushing, and screening to provide direct lump feed without the requirement for further processing (Jankovic, 2022). For low grade iron ores however, further beneficiation methods must be employed to minimise the amount of gangue minerals present and liberate the ore (Sahin, 2020). Methods available for beneficiation include gravity concentration, magnetic separation, floatation, bioleaching, selective flocculation, and reduction roasting followed by agglomeration (sintering/pelletizing) (Bulayani *et al.*, 2024). These upgrading processes exploit differences in the basic properties such as size, density, magnetic susceptibility, hydrophobicity to separate iron value from the gangue. Fig. 2 shows conventional methods employed in the beneficiation of iron ore.

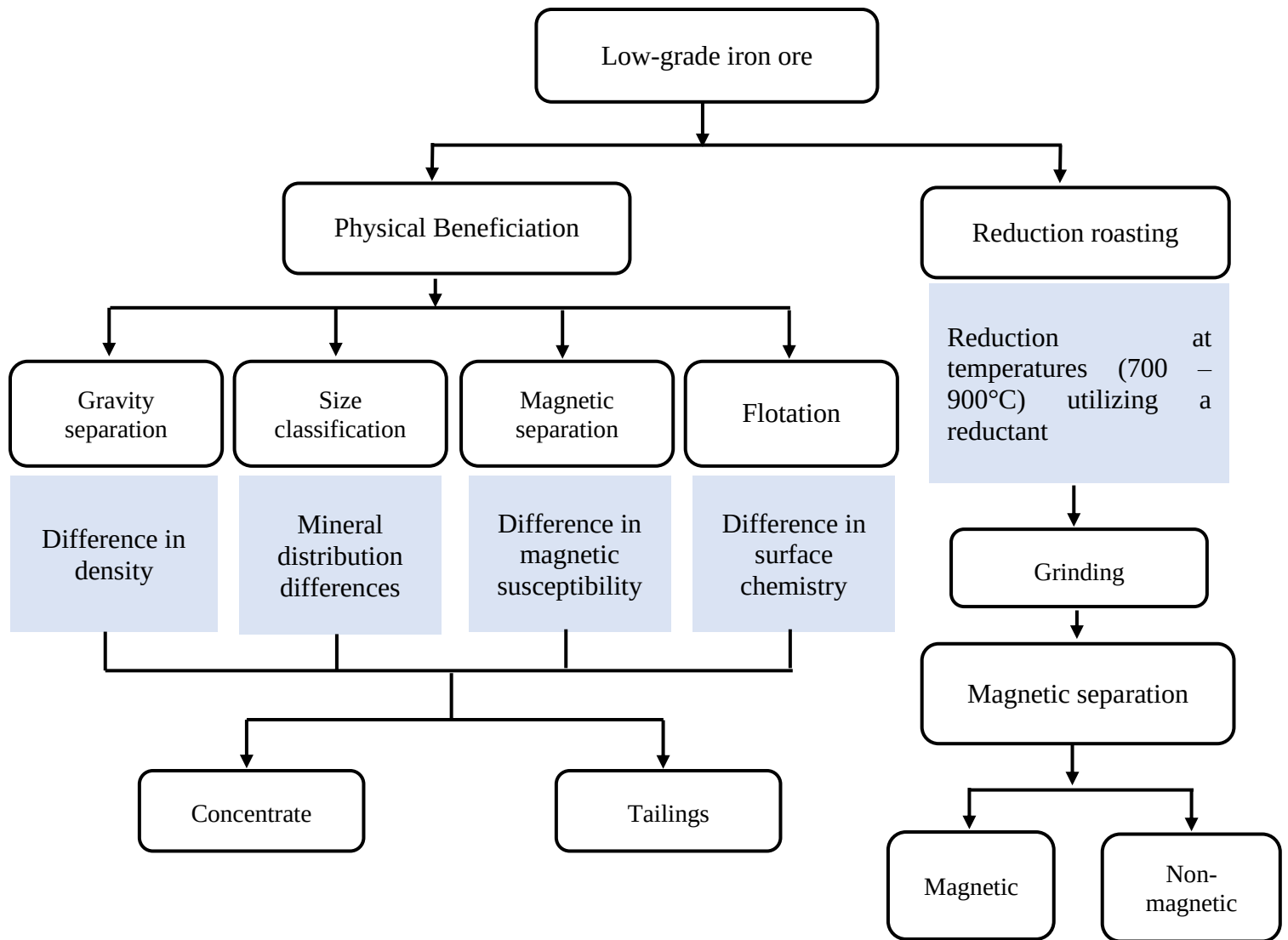


Figure 2. Conventional low grade iron ore beneficiation technique

### 2.6.1 Comminution

The initial step in iron ore processing is primary crushing. Large, run-of-mine (ROM) iron ore lumps, typically measuring 1-1.5 metres in diameter, are reduced in size to approximately 10-20 centimetres for high-grade iron ores. Lump product is preferred for rich ores as it attracts competitive prices compared to fine product (Jankovic, 2015). Primary crushing can be achieved using either jaw crushers or gyratory crushers, the selection of the primary crushing method can

be based on factors such as the iron ore grade, hardness and mineralogical composition. Primary crushing takes place in an open circuit and utilizes a dry feed process. This initial crushing stage reduces the large, run-of-mine (ROM) iron ore pieces down to sizes that are suitable for conveyor belt transportation and subsequent secondary crushing (Wills and Finch, 2016a). Any resident clay, tramp metal and wood are also removed at this stage as they can cause damage to downstream process units.

For the secondary crushing stage in iron ore processing, cone crushers are commonly used. These secondary crushers are typically smaller and lighter than the primary jaw crushers employed in the initial size reduction. Furthermore, impact crushers and roll crushers are occasionally employed as secondary crushing media, especially for softer and more brittle types of iron ore. (Haldar, 2018). The material output from the primary crushing stage is fed into the secondary crushing units for further size reduction. This product has a diameter of about 15cm and is more convenient to handle since most of the undesirable constituents have been removed in the first stage of crushing. The secondary crushing stage utilizes a dry feed process and produces material within the size range of 3-5 centimetres. This crushed material is now suitable for the next grinding step, if the secondary crushing is performed in an open circuit configuration.

For beneficiation of low-grade iron ores, a tertiary grinding stage is often required to improve mineral liberation and increase ore concentration. This further size reduction is usually accomplished using rod mills and ball mills, or a combination of the two. Jankovic et al (Jankovic, 2015) noted that a cone crusher can serve as a tertiary crusher when integrated into a closed circuit system, connecting the secondary crusher with a ball mill or rod mill. These mills can further decrease the feed particle size from approximately 5 cm to the ideal liberation size of 40 to 300  $\mu\text{m}$  (Wills and Finch, 2016b). Rod mills are designed to grind larger particles preferentially, which helps avoid the overgrinding of softer particles, leading to a smaller quantity of excessively fine ground material (Haldar, 2018).

Autogenous (AG) and semi-autogenous (SAG) mills are commonly used in many processing plants for grinding tasks (Gupta and Yan, 2006). Autogenous mills reduce ore size through the self-grinding action of large ore particles, without the addition of any grinding media in the mill. As a

result, the mill operates at a comparatively lower cost. However, variations in the hardness and abrasiveness of the ore can lead to inconsistencies in the final product. This is due to changes in the ore's physical breakage characteristics during the grinding process. In semi-autogenous grinding mills, steel balls are added along with the iron ore feed. The steel balls aid the action of the larger ore particles in pulverizing and further reducing the size of the ore (Haldar, 2018).

Fig. 3 illustrates a combination of size reduction techniques employed to achieve effective mineral liberation prior to the ore being subjected to the flotation process.

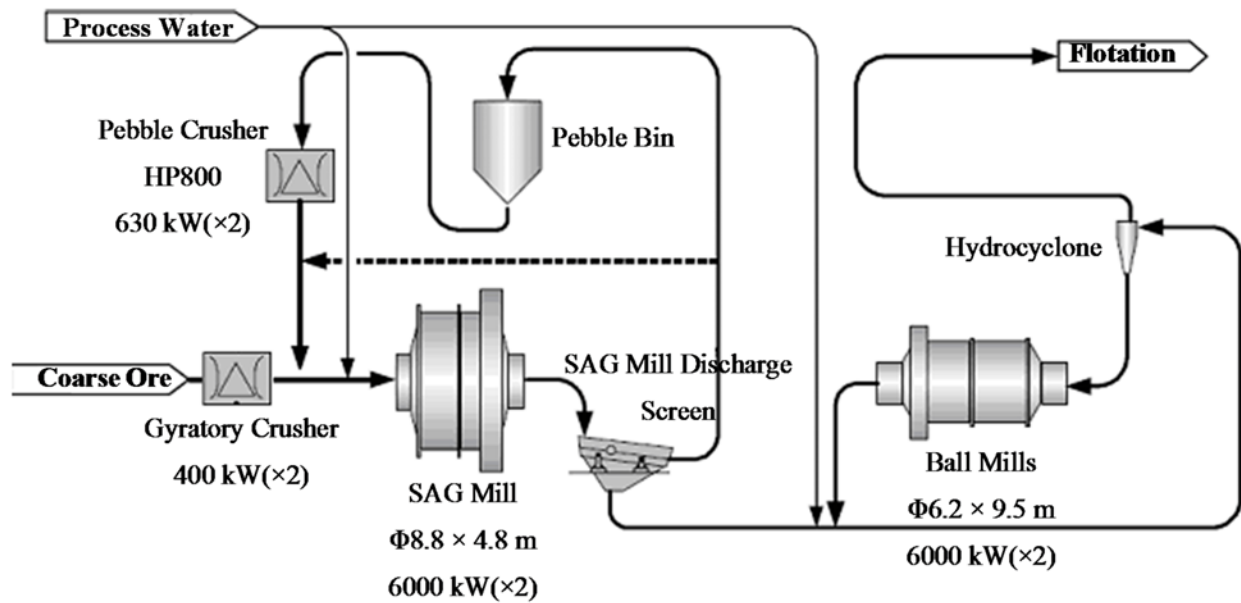


Figure 3. A combination of size reduction methods used prior to the flotation process. (Campos *et al.*, 2021).

Since iron ore encompasses varied chemical composition and mineralogy this allows for varied comminution response and liberation characteristics. The energy consumption required ultimately varies depending on the work indices of the minerals present (Campos *et al.*, 2021). Table 2 presents the various bond work indices for common minerals associated with iron ores.

Table 2. Minerals linked to iron ores and their corresponding Bond work index. ((‘Table of Bond Work Index by Minerals’, 2015; Bulayani *et al.*, 2024)).

| <b>Mineral</b>   | <b>Bond Work Index<br/>(kWh/t)</b> |
|------------------|------------------------------------|
| <b>Hematite</b>  | 12–14                              |
| <b>Magnetite</b> | 11–12                              |
| <b>Goethite</b>  | 10                                 |
| <b>Quartz</b>    | 13–15                              |
| <b>Alumina</b>   | 17–18                              |

During iron ore size reduction, the rate at which the size reduction process occurs is referred to as the kinetics of comminution. This includes comprehending how rapidly the ore particles break into smaller pieces when forces are applied. The kinetics of iron ore comminution are impacted by dissimilar factors such as crushing and grinding mechanisms, particle size distribution and ore properties.

During comminution of ores processes particle size distribution(PSD) is a critical factor, more especially in low grade iron ores (Bahrami, Farajzadeh and Kazemi, 2021). PSD analysis of ores is essential in grinding process control and helps in determination of cut sizes for other beneficiation techniques efficiency (Hanumanthappa *et al.*, 2020). For the desired cut size to be obtained grinding parameters including feed rate, mill speed and grinding media size are optimized(Kanda and Kotake, 2007; Matsanga, Nheta and Chimwani, 2023).

### **2.6.2 Gravity separation**

Gravity separation processes exploit the difference in density between two minerals as a simple method of achieving concentration (Sahin, 2020). In the case of haematitic ores, the iron oxide minerals present are typically denser than the predominantly siliceous gangue minerals found in

the ore (Helen, 2019; Nayak, Jena and Mandre, 2021). Table 3 presents the specific gravity values for commonly found iron-bearing minerals.

*Table 3. Specific gravity of iron-bearing minerals(Sahin, 2020; Bulayani et al., 2024)*

| <b>Name of Ore</b> | <b>Fe content (%)</b> | <b>Specific Gravity</b> |
|--------------------|-----------------------|-------------------------|
| Hematite           | 70.0                  | 5.00–5.30               |
| Magnetite          | 72.4                  | 5.17–5.18               |
| Goethite           | 62.5                  | 3.96                    |
| Limonite           | 60.0                  | 2.70–4.30               |
| Siderite           | 48.3                  | 3.85                    |
| Iron pyrite        | 46.6                  | 4.90–5.20               |

Jigging is an old gravity separation technique that is widely used in iron ore processing and has undergone decades of technological improvements. In a basic jigging process, a pulsating fluid flows through a layer of ore particles, leading to the stratification of lighter and heavier particles according to their specific gravity. The heavier particles accumulate in the lower layer of the bed (Gupta and Yan, 2006; Gomes, Tomi and Assis, 2015). Jigging process is described(Mukherjee, Bhattacharjee and Mishra, 2006; Naudé *et al.*, 2013; Rachappa, Prakash, and Amit, 2015) as having low operational costs and therefore suitable for upgrading low-grade ores to produce a saleable product. Contemporary jigs are engineered to function according to the size and properties of the ore being treated, and can achieve satisfactory separation down to particles as small as 75  $\mu\text{m}$ (Murthy, Tripathy and Kumar, 2011). Fig. 4 illustrates a jigging technique working principle (Ambrós, 2020). South Africa's Sishen iron ore mine, situated in the Northern Cape Province, is one of the largest open-pit iron ore mines in the world. The Sishen mine uses jigging technology to beneficiate run-of-mine material that would otherwise be regarded as waste, producing a saleable concentrate with an iron content of 64% (Naudé *et al.*, 2013).

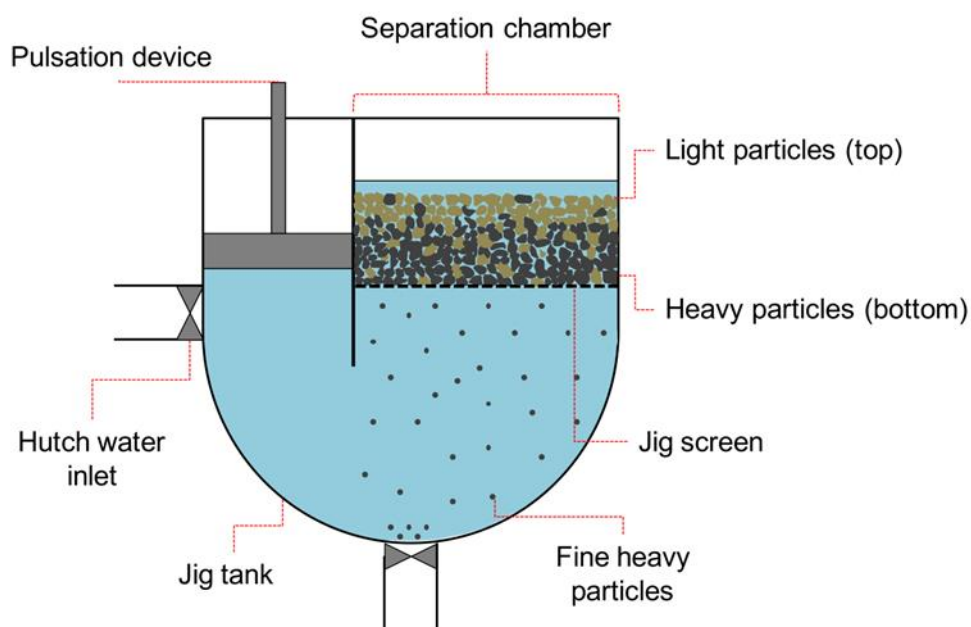


Figure 4. The fundamental principle of a typical jigging process.

Spiral concentrators are used all over the world to produce mineral concentrates, including during treatment of iron ore fines. The basic principle of a spiral as a flowing film gravity separator is that it uses a combination of gravity and fluid-related dynamic forces to effect separation of fine heavy minerals from light counterparts (van Dam *et al.*, 2014). Conventional spirals are reported (Sadeghi, Bazin and Renaud, 2016; Wills and Finch, 2016c) to typically operate within a size range of 1 to 3 mm down to 75  $\mu\text{m}$ . Although high separation efficiency is necessary for iron ore fines, significant advancements have been made in enhancing the performance of spiral concentration for the beneficiation of iron ore (Boucher *et al.*, 2016; Grobler, Naudé and Zietsman, 2016; Sadeghi, Bazin and Renaud, 2016; Nienaber and Auret, 2019). Spirals have a widespread application in concentrating iron ore fines of 3mm to 45 $\mu\text{m}$  size range due to higher efficiency at this size range compared to other conventional gravity concentrators like jigs and shaking tables (Rath and Singh, 2007; Gülsoy and Gülcan, 2019). Spirals typically operate at pulp density between 15 – 45 % solids by weight. Iron ore fines beneficiation plants frequently utilize multi-stage spiral circuits, including rougher, scavenger, and cleaner stages, to obtain an upgraded

product with high recovery rates. The spiral concentration output can be further refined using magnetic separation, which increases the overall iron content grade, resulting in a marketable product (Jordens *et al.*, 2014). A recently developed flowing film concentration method known as the multi-gravity separator (MGS) that uses shaking and centrifugal force to achieve separation. The principle of the MGS is based on a conventional shaking table, only with the horizontal concentrating surface rolled into a cylindrical drum which is then rotated (Gupta and Yan, 2006). It is designed for treating fine and ultra-fine material with a maximum particle size of 0.5mm, down to 1  $\mu\text{m}$  which would otherwise be lost to the tailings when using conventional gravity concentrators like jigs and spirals (Roy, 2009). The MGS is particularly important for processing iron ore slimes that contain very low levels of iron and high amounts of silica and alumina.

### 2.6.3 Electrostatic separation

Electrostatic separation is not widely applicable to iron ores because it depends on variations in electrical conductivity between iron oxides and gangue minerals. It is most effective on crystalline, non-magnetic iron oxides that are finer than 1.7 mm and coarser than 75  $\mu\text{m}$ , provided the mineral surfaces are clear of slime or dust coatings (DeVaney, 1985).

### 2.6.4 Flotation

The selective separation of minerals via flotation relies on the natural and induced chemical and physical properties of the mineral surfaces to facilitate separation (Zhang *et al.*, 2021). The flotation process for mineral separation has become well-regarded due to its ability to handle ores

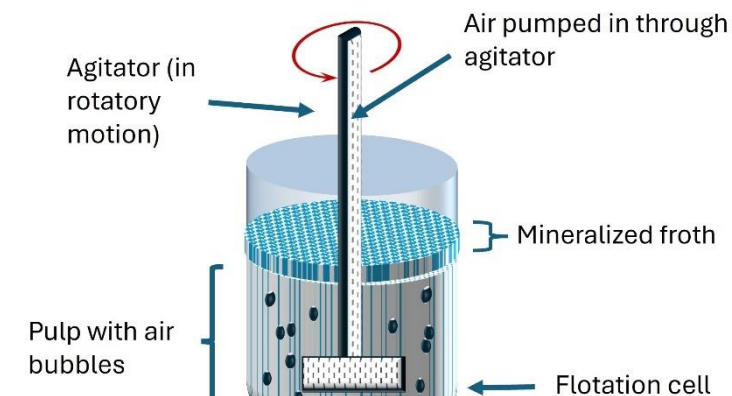


Figure 5. Froth flotation principle

with complex mineral compositions, low-grade ores with density variations, or particle sizes too small for effective gravity separation(Zhang *et al.*, 2021). The recovery of materials from the pulp through flotation involves three mechanisms: the selective adherence of desired mineral particles to air bubbles, their capture in the water flowing through the froth, along with aggregation of particles within the froth phase(Araujo, Viana and Peres, 2005; Batchelor *et al.*, 2016). The flotation separation mechanism is shown in Fig. 5.

Iron ore flotation operations can either be of anionic or cationic types. In direct anionic flotation, fine-sized iron oxide values (e.g., siderite, hematite) are floated preferentially from silica minerals (usually quartz)(Nakhaei and Irannajad, 2018). For reverse cationic flotation rather, the siliceous gangue material is floated, the iron values are suppressed and collected as the underflow(Faith Okoro, Damilola Femi-Oyewole, and Opeyemi Okunlola, 2016). Reverse cationic flotation is the most used flotation method for iron ore. For iron ores consisting predominantly of hematite and quartz (BHQ), corn starch is frequently employed as a depressant for hematite, while ether amine blends are used as collectors for quartz. (Filippov, Severov and Filippova, 2014). Examples of reagents used in reverse cationic flotation include ether amines, dodecyl amine, di-amines and quaternary ammonium salts (Nakhaei and Irannajad, 2018). Table 4 provides a comparison between direct and reverse flotation.

Flotation studies reflect that soluble starches are the most efficient starches for depressing hematite. Nonetheless, the overall efficiency of the flotation process greatly depends on the mineral liberation characteristics and the type of gangue material involved (Araujo, Viana and Peres, 2005; Pattanaik and Venugopal, 2018; Yang, 2018). The presence of slimes in flotation results in higher reagent consumption and decreased flotation efficiency. Therefore, de-sliming is a crucial step for achieving effective separation in iron ore flotation (Carpenter, Iveson and Galvin, 2019). Table 4 contrasts and distinguishes between direct and indirect flotation (Knights *et al.*, 2012; Cao, Zhang and Cao, 2013; Filippov, Severov and Filippova, 2014; Zhang *et al.*, 2021).

Iron ore flotation is predominantly carried out in India, China, and Brazil. Process control plays a crucial role in flotation operations. In South Africa, Mintek created a sophisticated system for

stabilizing and optimizing flotation, which was later implemented at Vale's Cauê iron ore processing facility in Brazil (Knights *et al.*, 2012). The performance analysis of the study revealed a 2.7% increase in iron recovery and a 1.2% reduction in the grade of iron tailings.

*Table 4. Comparison of direct and reverse flotation.*

| <b>Component</b>                                                                            | <b>Direct Flotation</b>                                          | <b>Reverse Flotation</b>                                             |
|---------------------------------------------------------------------------------------------|------------------------------------------------------------------|----------------------------------------------------------------------|
| Objective                                                                                   | Floats iron-bearing oxides                                       | Floats gangue minerals and depresses iron oxides                     |
| Reagents                                                                                    | Anionic reagents (fatty acids, petroleum sulfonates)             | Cationic reagents (primary amines, quaternary ammonium salt)         |
| pH modifiers (e.g. sulphuric acid calcium hydroxide, sodium carbonate,)                     | Establishes optimal environment for the flotation of iron oxides | Establishes optimal environment for the flotation of gangue minerals |
| Depressants (e.g. starch, sodium silicate)                                                  | Depress unwanted minerals to avoid their flotation               | Depress iron oxides to avoid their flotation                         |
| Activators (e.g. lead nitrate copper sulphate)                                              | Not used                                                         | Applied before adding collectors to stimulate gangue minerals        |
| Collectors (e.g. sodium dodecyl, dodecanoic acid, amines, fatty acids, xanthates, sulphate) | Increase iron oxides hydrophobicity                              | Increase the hydrophobicity of gangue minerals.                      |

### **2.6.5 Reduction roasting and magnetic separation**

Reduction roasting, referred to as carbothermal reduction, is a developing method for processing low-grade iron ores. This technique involves roasting hematite, goethite, and siderite in a reducing environment, resulting in the production of magnetite concentrates that can be easily separated using Low-Intensity Magnetic Separation. (LIMS)(Rath *et al.*, 2014). Fig. 6 illustrates a typical schematic flowchart for the reduction roasting of iron ore, integrated with magnetic separation.

In iron ore roasting, a reducing atmosphere is typically created using carbonaceous reduction complexes like coal, sawdust, and biomass. (Rayapudi, Agrawal and Dhawan, 2020).

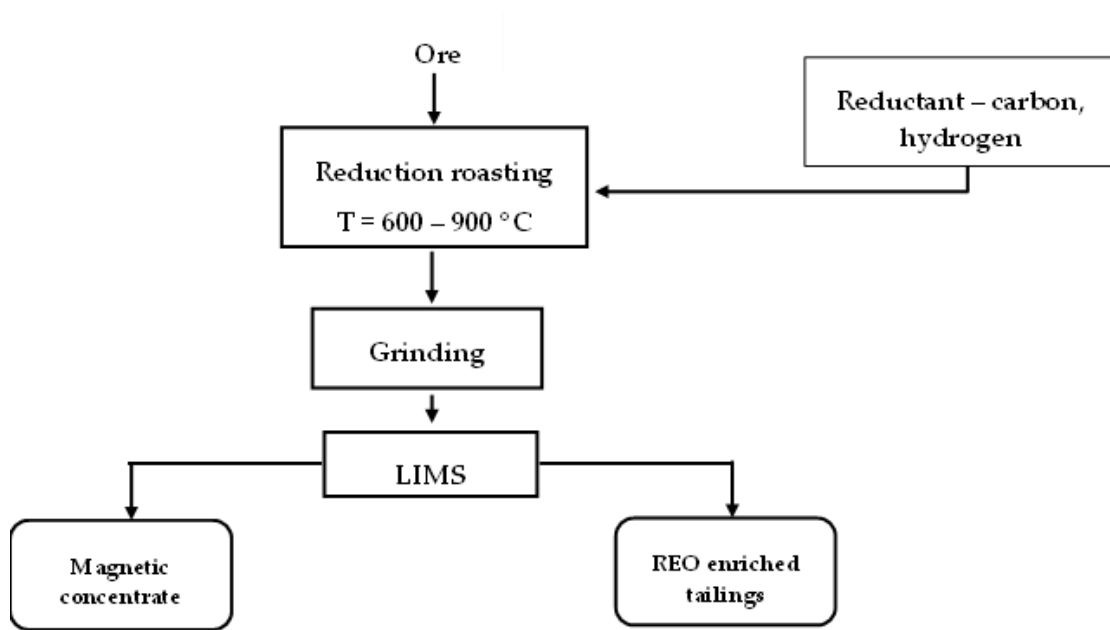
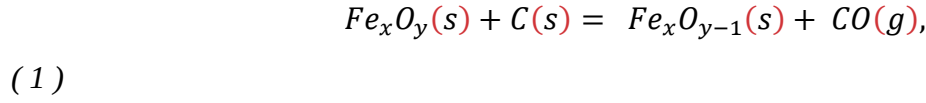


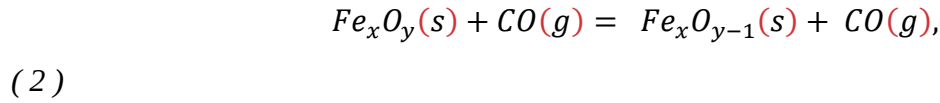
Figure 6.. illustrating iron ore reduction roasting combined with magnetic separation

Some researchers have observed that the reduction roasting process can convert weakly magnetic minerals into magnetic ones by roasting them in a reducing atmosphere at elevated temperatures, typically  $\geq 600$  °C (Faris *et al.*, 2017; Rayapudi, Agrawal and Dhawan, 2020; Panda *et al.*, 2023; Baqer and Hafiz, 2024). Roasting iron ore at excessively high temperatures is not recommended, as it can result in particle sintering or, in certain instances, the creation of paramagnetic wustite (Sahu *et al.*, 2019). Researchers have noted that the reduction roasting of iron ore involves three specific phase transformations: (a) hematite is converted to magnetite at temperatures between 200 and 250 °C; (b) magnetite is subsequently reduced to wustite within the range of 500 to 900 °C; and (c) finally, wustite is transformed into iron metal at temperatures from 900 to 1300 °C (Stephens, Langston and Richardson, 1953; IWASAKI and PRASAD, 1989; Rath *et al.*, 2016). The reduction of iron ore using a reducing agent such as coal involves several stages: (a) the diffusion of the reducing gas through the boundary layer; (b) interface reduction through intra-particle diffusion; (c) the transfer of  $\text{Fe}^{2+}$  ions and electrons to the iron core; (d) interface oxidation via intra-particle diffusion; and (e) the diffusion of the oxidizing gas across the boundary layer.

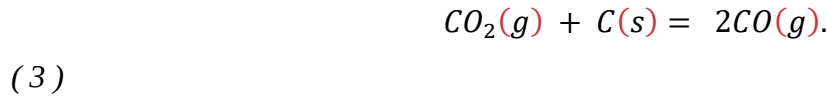
The reduction reactions that take place during the roasting of iron ore with solid carbon can be outlined as follows (Sahu *et al.*, 2019):



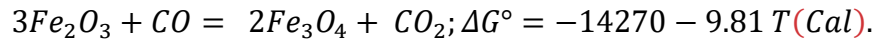
Where, x = 1, 2, or 3,



Where, y = 1, 3, or 4,



Ultimately, the overall reaction for the conversion of hematite to magnetite during reduction roasting with a reducing agent, such as carbon or carbon monoxide(C or CO) can be expressed as follows:



The reaction kinetics of iron ore reduction pertains to the rate at which iron oxide is converted into metallic iron by eliminating oxygen. The speed of reduction influences production rates, which subsequently affects the economic feasibility and competitiveness of the processing technology (El-Husseiny *et al.*, 2015). Fig. 7 illustrates iron ore reduction through CO reaction kinetics.

Reduction kinetics are usually characterized by the correlation between the degree of reduction of iron oxides and the time taken for the reduction process (Guo *et al.*, 2017a). The degree of reduction ( $\alpha$ ) is calculated using the following equation:

$$\alpha = \frac{W_{Lt}}{W_L} = \frac{m_0 - m_t}{m_0 + m_c + m_v} \quad (4)$$

In this context,  $\alpha$  represents the degree of reduction of the iron oxide;  $W_{Lt}$  refers to the weight loss of the sample (a homogeneous mixture of iron ore and coal) at time t, determined by the difference

between the initial weight ( $m_0$ ) and the weight at time ( $m_t$ ); and WL indicates the theoretical weight loss during the reduction, which encompasses the total mass of the oxygen atoms in the iron-bearing compound ( $m_O$ ), coal fixed carbon ( $m_C$ ), and coal volatile matter ( $m_V$ ).

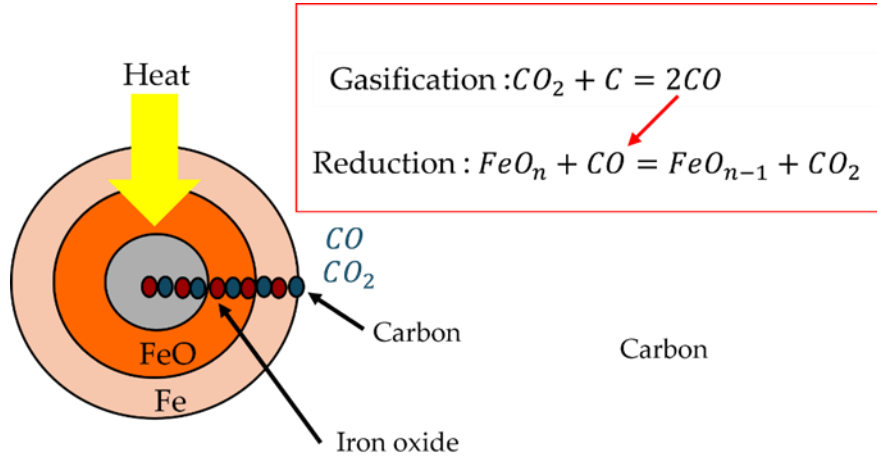


Figure 7. showing reduction of iron ore through CO kinetics

To assess the reduction of iron-bearing compounds, a single restricted-link kinetic model is typically employed. When the rate-limiting step of the reduction reaction involves the interfacial chemical reaction, the kinetic model can be expressed by the following equation (Lu *et al.*, 2016):

$$1 - (1 - \alpha)^{\frac{1}{3}} = kt$$

(5)

When the gasification of carbon serves as the rate-limiting step in the reduction reaction, the kinetic model is expressed by the following equation:

$$\ln(1 - \alpha) = -kt$$

(6)

When gas diffusion through the particles is the rate-limiting step in the reduction reaction, the kinetic model can be outlined as follows:

$$\left[1 - (1 - \alpha)^{\frac{1}{3}}\right] = kt$$

(7)

k signifies the reaction constant, t indicates time, and  $\alpha$  represents the degree of reduction.

The activation energy of the reaction can be calculated using the Arrhenius Equation, expressed as follows (Zhang *et al.*, 2020):

$$k = A \exp\left(-\frac{E\alpha}{RT}\right)$$

(8)

By taking the logarithm of Equation (8), the Arrhenius Equation can be represented in its linear form as follows:

$$\ln k = -\frac{E\alpha}{R} \times \frac{1}{T} + \ln A$$

(9)

Here, A represents the pre-exponential factor,  $s^{-1}$ ;  $E_{\alpha}$  denotes the activation energy reaction,  $J \cdot mol^{-1}$ ; R is the ideal gas constant  $8.314 J \cdot mol^{-1} \cdot K^{-1}$ ; T is the temperature, K.

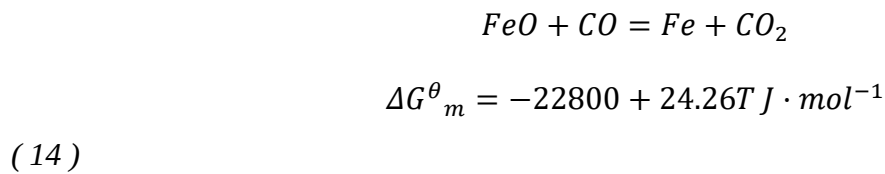
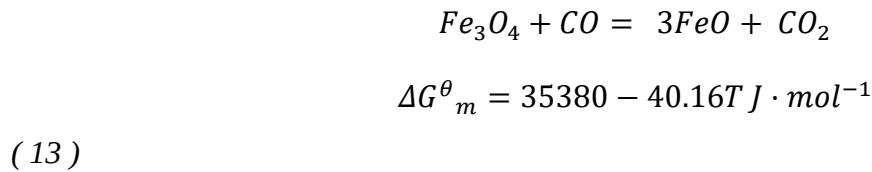
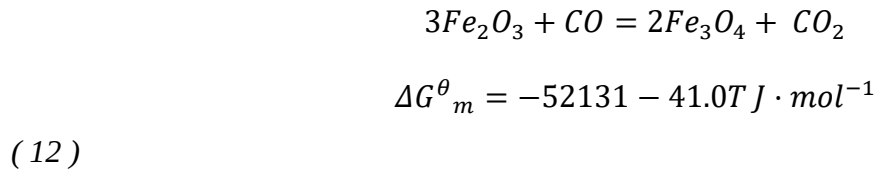
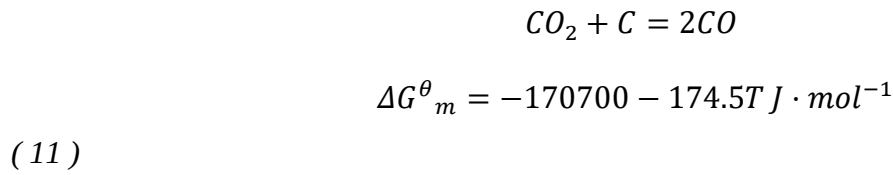
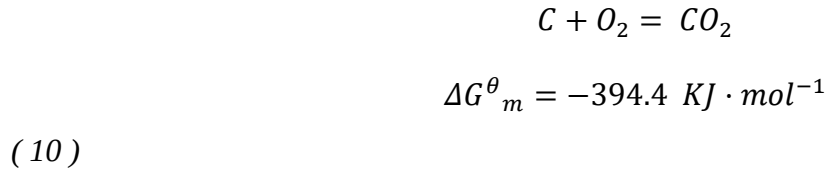
The Arrhenius equation links the pre-exponential factor (A), which represents the frequency of successful collisions between reactant particles, with the absolute temperature and the rate constant of a chemical reaction (Lu *et al.*, 2016; Mishra, 2020). It provides insight into how reaction rates are influenced by absolute temperature.

The thermodynamics of iron oxide reduction mainly focuses on the equilibrium that is established between the iron oxides and the reducing agents employed (Ghassemi and Shahidian, 2017a). Thermodynamic analysis determines whether a reaction is feasible and whether it will proceed in the forward direction. The viability of the reaction is signified by a decrease in free energy. The fundamental principles of the thermodynamics involved in iron ore reduction were first presented by Dannatt and Ellingham (Dannatt and Ellingham, 1948), who developed a graphical method illustrating the standard free energy of oxide formation as a function of temperature. This approach

is crucial for understanding the stability of compounds at various temperatures (Roy, Nayak and Rath, 2020a).

In the reduction roasting process, the primary reactions include the gasification of carbon and the reduction of iron-containing compounds (Cao *et al.*, 2016; Guo *et al.*, 2017b).

Various fundamental chemical reactions in the Fe-O-C system (Han *et al.*, 2015).



The standard Gibbs free energy change,  $\Delta G$ , indicates the thermodynamic favourability of a physical or chemical process. Often referred to as Gibbs function or free enthalpy, Gibbs free energy is a thermodynamic potential that measures the maximum reversible work a system is capable of performing under constant temperature and pressure conditions (Ghassemi and Shahidian, 2017b).

### 2.6.6 Magnetic separation

Magnetic separation exploits magnetic susceptibility differences between valuable minerals and gangue minerals in an ore deposit to concentrate the desired material (Chen and Xiong, 2015). Magnetic separators are categorized based on the strength of their magnetic field into two types: low-intensity and high-intensity (Xiong, Lu and Holmes, 2015). For instance, the Kiruna iron ore operations in Sweden use low intensity separators(LIMS) to process low-phosphorus magnetite ore that contains more than 60% iron (Dwari, Rao and Reddy, 2013).

Magnetic separation technique procedures are dependent on various processing parameters including mineral composition, magnetic susceptibility and liberation size(Vapur *et al.*, 2020). Both low and high-intensity magnetic separators are capable in operating on both dry and wet feed basis and these equipment exploit magnetic susceptibility differences(Chen and Xiong, 2015). Fig. 8 illustrates how a wet based low intensity drum separator works.

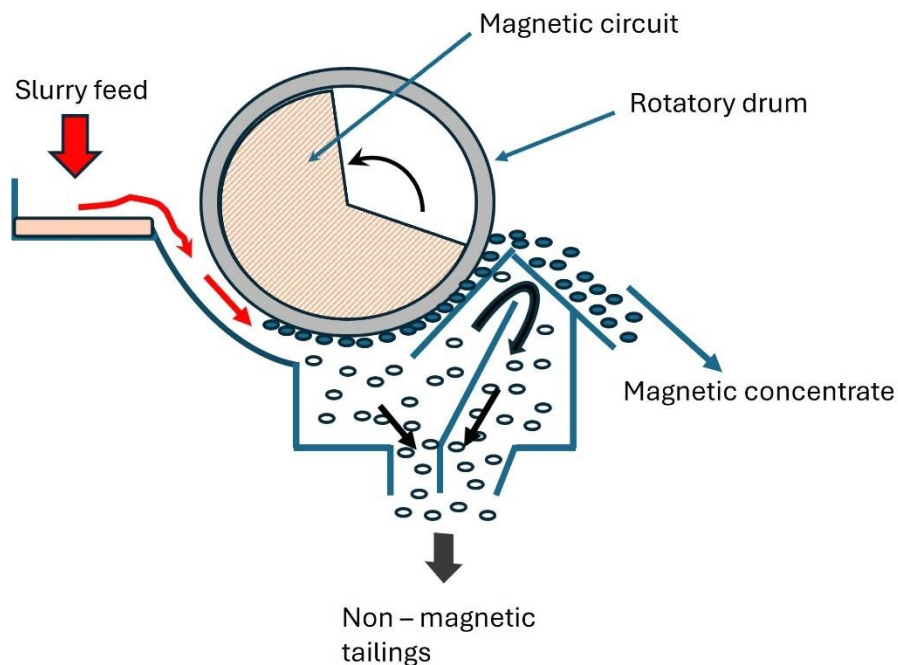


Figure 8. Drum magnetic separator

LIMS equipment functions with magnetic fields ranging from 1000 to 3000 gauss, while HIMS equipment operates with much stronger magnetic fields, reaching up to 20,000 gauss (Sahin, 2020). Minerals exhibit different magnetic susceptibilities degrees and this aids in separation since only paramagnetic and ferromagnetic minerals are attracted meanwhile diamagnetic materials are repelled (Rosenblum and Brownfield, 2000; *Something You Should Know About Magnetic Drum Separator* | *Stanford Magnets*, no date). Table 5 presents common iron oxides and their magnetic susceptibilities degrees.

Table 5. Magnetic susceptibilities of minerals containing iron (Bulayani et al., 2024)

| Minerals                                                             | Magnetic Susceptibility | Fe Content (%) |
|----------------------------------------------------------------------|-------------------------|----------------|
| Magnetite                                                            | 1156                    | 72.4           |
| Vanadic titano-magnetite ( $\text{TiO}_2$ , $\text{V}_2\text{O}_5$ ) | 917                     | 63.7           |
| Hematite ( $\text{Fe}_2\text{O}_3$ )                                 | 0.6–2.16                | 69.94          |
| Specularite ( $\text{Fe}_2\text{O}_3$ )                              | 3.7                     | 69.94          |
| Limonite ( $\text{mFe}_2\text{O}_3 \cdot \text{nH}_2\text{O}$ )      | 0.31–1                  | 48–62.9        |
| Siderite ( $\text{FeCO}_3$ )                                         | 0.7–1.5                 | 48.3           |
| Chromite ( $\text{FeO} \cdot \text{Cr}_2\text{O}_3$ )                | 0.62–0.89               | 25             |
| Pyrrhotite ( $\text{Fe}_7\text{S}_8$ )                               | 57                      | 63.53          |
| Ilmenite ( $\text{FeTiO}_3$ )                                        | 0.35–5                  | 33.33          |
| Wolframite ( $(\text{Fe}, \text{Mn})\text{WO}_4$ )                   | 0.5                     | 15.60          |

More than 90% of global iron ore production is treated through magnetic separation, since the main iron ore minerals are predominantly paramagnetic or ferromagnetic (Wills and Finch, 2016c; Roy, Nayak and Rath, 2020a). The white gangue minerals in iron ore viz. quartz, feldspar and calcite have magnetic susceptibilities of close to zero, making their magnetic separation from iron-

bearing minerals relatively easy (Rosenblum and Brownfield, 2000; Sahu *et al.*, 2019). Among iron minerals, magnetite is the primary ferromagnetic mineral that is concentrated using low-intensity magnetic separators ( $< \sim 0.3$  T) due to stronger magnetic properties (Wills and Finch, 2016c). Wet LIMS is typically used for smaller particle sizes (below 5mm) as it produces less dust, hence reducing mineral losses. In contrast, hematite is a weakly paramagnetic mineral and can only be efficiently separated using high-intensity magnetic separators with a field strength of 2T or more (Ezhov and Shvaljov, 2015). Wet high-intensity magnetic separators (WHIMS) are commonly employed for this application, processing iron ore slurries containing fine particles of sizes less than 75  $\mu\text{m}$  (Muthaphuli, 2014).

#### **2.6.7 Processing Botswana Iron Ores**

The processing of Botswana's iron ores, particularly from the Ikongwe Iron Ore Mine, can be effectively achieved using various techniques discussed in this study. Table 6 provides a summary of potential conventional processing techniques alongside their advantages and limitations.

Table 6. Conventional iron ore processing techniques including their pros and cons(Bulayani et al., 2024).

| Processing technique | Pros                                                                                                                                                                                                                                                                                         | Cons                                                                                                                                                                                                                                                                                                                                                   |
|----------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Gravity separation   | <ul style="list-style-type: none"> <li>• Low cost</li> <li>• Environmentally friendly (no chemicals required)</li> </ul>                                                                                                                                                                     | <ul style="list-style-type: none"> <li>• Less effective for finer ores</li> <li>• Low efficiency for ores with close density differences (less disparity).</li> </ul>                                                                                                                                                                                  |
| Magnetic separation  | <ul style="list-style-type: none"> <li>• High efficiency for magnetite ores</li> <li>• Can handle large volumes</li> <li>• Environmentally friendly</li> <li>• Conducted in both dry and wet environments</li> <li>• Versatile and can separate iron ore into multiple components</li> </ul> | <ul style="list-style-type: none"> <li>• Less effective for ores with weak magnetic properties</li> <li>• Magnetic fields generation requires significant energy input</li> <li>• Less efficient in finer dispersed ore particles</li> </ul>                                                                                                           |
| Reduction roasting   | <ul style="list-style-type: none"> <li>• Effective for converting and recovering high-grade iron value from low-grade resources or finer iron ore tailings.</li> <li>• Effective for complex and low-grade ores</li> </ul>                                                                   | <ul style="list-style-type: none"> <li>• The process requires precise control of temperature, time, and reductant dosage</li> <li>• High energy consumption due to roasting</li> </ul>                                                                                                                                                                 |
| Flotation            | <ul style="list-style-type: none"> <li>• Effective for the treatment of carbonate-rich iron ores</li> <li>• Allows for the recovery of fine particles that maybe lost in other separation methods</li> </ul>                                                                                 | <ul style="list-style-type: none"> <li>• Complex process, requiring careful control of various factors like pH, reagent type, and dosage</li> <li>• De-sliming is necessary to enable effective flotation, which doubles process complexity and operating cost</li> <li>• Not as effective for ores with high levels of impurities or mixed</li> </ul> |

Reduction roasting combined with magnetic separation was chosen as the preferred method for processing Botswana ores due to its effectiveness in handling ultra-fine particles and complex mineralogy, enabling the removal of finely disseminated impurities that are difficult to eliminate by other methods. This combination optimizes the separation index by converting hematite to magnetite, achieving higher iron recovery rates compared to traditional techniques such as gravity separation or flotation. The process is energy-efficient, operating at moderate temperatures with reduced energy consumption, and is flexible across a wide range of ore compositions, making it particularly suitable for Botswana's ore characteristics. Additionally, it utilizes locally abundant semi-bituminous coal as a reductant, which is both economically viable and environmentally friendly, producing fewer tailings and minimizing pollution. These advantages, along with the availability of local raw materials, make this combination of reduction roasting and magnetic separation the optimal and sustainable choice for beneficiation of Botswana's iron ores.

## CHAPTER 3

This chapter focuses into the iron ore beneficiation process utilizing coal as a reductant in a muffle furnace, focusing on the conversion of hematite to magnetite. The methodology includes the characterisation of local iron ore and coal samples, detailing their mineralogical and chemical properties. Taguchi experimental design, optimisation of reduction roasting parameters and validation of the effectiveness of reduction roasting and magnetic separation techniques. The chapter also integrates kinetic and thermodynamic modelling approaches.

Iron ore beneficiation with coal as a reductant in a muffle furnace process entails converting hematite into magnetite, which is ferromagnetic. This magnetite can then be separated from non-metallic materials using a drum magnetic separator. One study by Liu et al [83] investigated the reduction of iron ore using coal in a muffle furnace. The researchers found that factors such as temperature, coal particle size, and the amount of coal used had a considerable impact on the efficiency of the reduction process. Later studies have documented successful carbothermic reduction of low-grade iron ores, which were subsequently recovered through magnetic separation [63], [69], [69], [76], [84]. Yu et al [85] analysed the reduction roasting and magnetic separation of hematite ore fines containing carbonates as gangue. Fig. 9 represents the proposed iron ore beneficiation approach strategies.

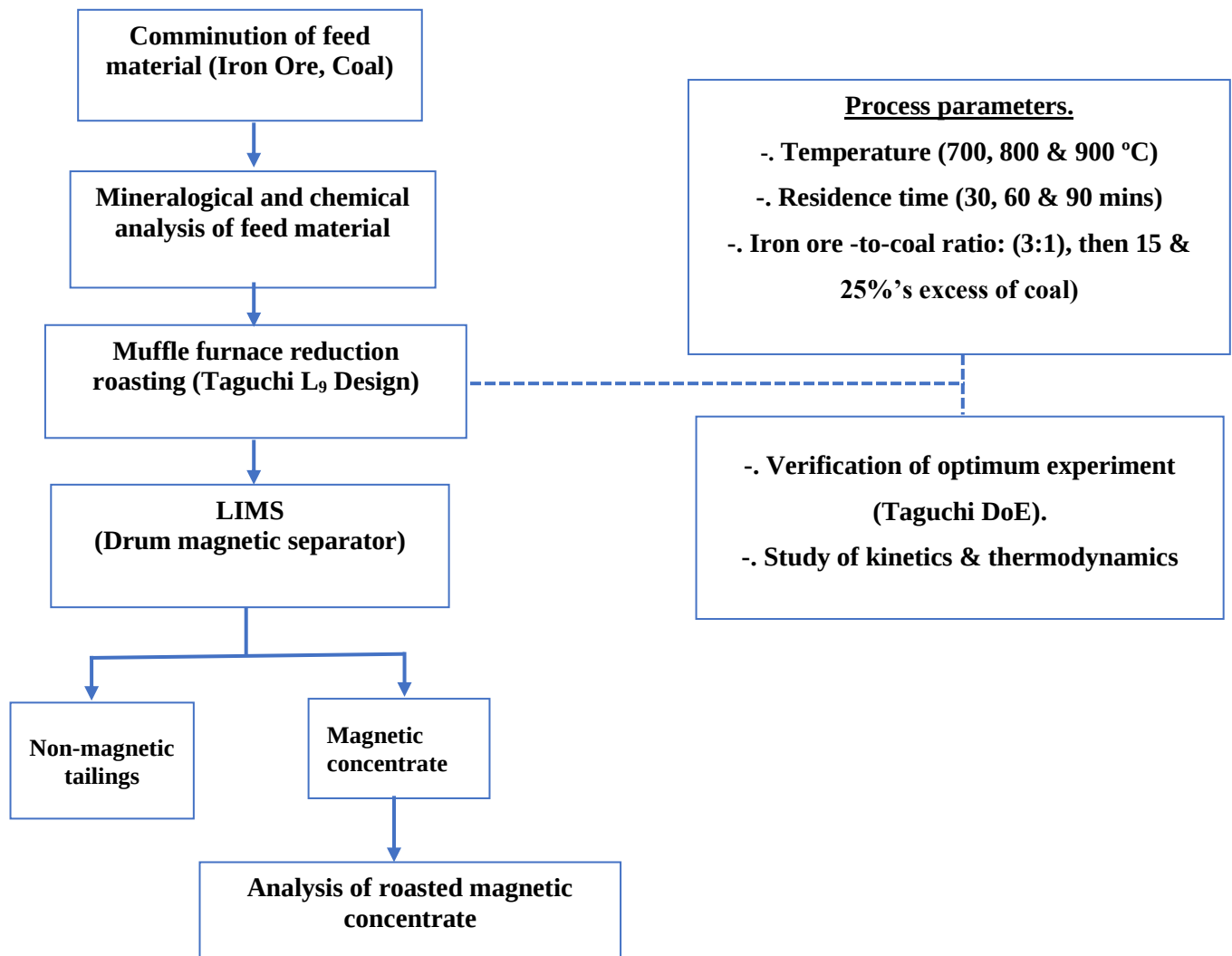


Figure 9. A schematic illustration of the proposed methodology

### 3.1 Characterization of local raw materials for mineralogy and chemistry

#### 3.1.1 Sample collection and preparation

The low-grade iron ore sample subjected to this study was collected from surface outcrops at the Ikongwe Iron Ore Mine near Shoshong village in the Central district of Botswana. The collected iron ore sample was initially rocks in nature and required size reduction to facilitate subsequent

mineralogical and chemical analysis. The iron ore rocks were initially crushed using a geological pick (rock pick) to reduce the particle size to approximately 15-20 mm in diameter. The iron ore lumps were primarily crushed using a jaw crusher to about 5-8 mm in diameter, subsequently the sample was then subjected to secondary crushing using a cone crusher, resulting in a finer particle size of approximately  $\pm 2$  mm in diameter. The secondary crushed iron ore material was ground using a ball mill until the iron ore sample reached a fineness of about 40-120  $\mu\text{m}$ . A sieve shaker with different sieve sizes was used to obtain fine particles passing through 80  $\mu\text{m}$ . Fig. 10 illustrates different iron ore aggregates as it was subjected through comminution techniques, whereas Fig. 11 shows equipment that was used. A rotary splitter was used to thoroughly mix and homogenize the finely ground iron ore sample in order to minimize the heterogeneity within the sample in order to have a representative and consistent sample to ensure reliability and accuracy of mineralogical and chemical analysis results. A quadruple sample divider was employed to split the pulverized samples into four smaller portions, each designated for different analyses.



*Figure 10. Size reduction from ROM to about 40-120 $\mu\text{m}$*



Figure 11. Equipment used in comminution of raw materials; a) Rock pick, b) Cone crusher, c) Ball mill

### 3.1.2 Non-coking coal preparation

Non-coking coal was collected from Morupule Coal Mine a local mine in Palapye, Botswana. The coal was obtained in larger aggregates. Therefore, it was primarily crushed using a jaw crusher from aggregates ranging between 10-15mm. For further crushing a mortar and pestle were used to ground primarily crushed coal obtaining particle sizes pass through 850  $\mu\text{m}$ . The Morupule coal

sample was then pulverized using a planetary ball mill running at 150 rpm and subsequently sieved to obtain a particle size fraction of 80–100  $\mu\text{m}$ . Fig. 12 illustrates coal size before and after size reduction.

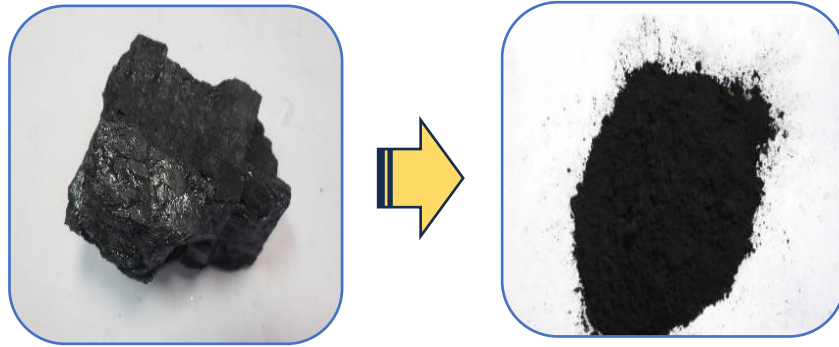


*Figure 12. Coal a)before and b)after size reduction*

A pulverized coal sample was gathered for thermogravimetric analysis (TGA) to assess fixed carbon content and other coal components. This is important because coal is a complex material with diverse properties influenced by its origin, rank, and age. Proximate analysis of non-coking Morupule coal was performed using a thermogravimetric analyser (TGA 701) following the ASTM D7582 MVA method for coal shown in Fig. 13. This analysis involved monitoring weight changes in the samples as they were heated under various conditions.



*Figure 13. Thermogravimetric analyser (TGA 701) used for proximate analysis*

### **3.1.3 Characterization.**

Physical and chemical compositions of iron ore influence the processing routes of that particular iron depending on the targeted properties. Mineralogical characterization techniques are employed to determine present major minerals and gangue minerals in an ore, their associations, and liberations (Dwari, Rao and Reddy, 2013; Krishna *et al.*, 2013). The as received iron ore sample was examined through XRD technique to assess its phase and microstructure, and Scanning Electron Microscopy (SEM) coupled with EDX to analyse its surface morphology (ore's surface texture, particle shape, and size distribution) and provides qualitative chemical composition estimates at specific points. This combination of techniques (SEM, XRF, and XRD) ensures that both the morphological and chemical properties of the iron ore are well characterized, providing a robust foundation for analysing the changes that occur during roasting. XRF analysis were employed on the raw iron ore for elemental composition analyses providing quick, accurate, and comprehensive data that supports efficient processing.

### **3.1.4 Chemical analysis**

Elemental composition analysis of feed iron ore is the first step in understanding its chemical composition particularly iron concentration which is vital in evaluating the efficiency of beneficiation methods and monitoring compositional alterations following beneficiation. This

analysis is also indispensable for quality control, as it identifies present impurities that could impair the efficacy of the product quality. Initial iron ore content knowledge is instrumental in tailoring reduction roasting experimental conditions. The elemental composition of ore sample was assessed through the X-Ray Fluorescence (XRF) analytical technique using a Rigaku ZXS Primus II instrument shown in Fig. 14.



*Figure 14. Rigaku ZXS II equipment.*

### **3.1.5 Mineralogical and morphological analysis**

Iron and its oxides have a crystalline structure, with atoms arranged in regular, repetitive patterns. When its exposed to an X-ray beam, these materials cause the beam to diffract, resulting in a measurable pattern of intense peaks (Morgan, 1990; Hessels, 2023). The phases of the pulverized iron ore sample were identified using diffraction data collected with a Bruker D8 Advance diffractometer (Fig. 15a). The analysis employed Cu K- $\alpha$  radiation with a wavelength of 1.540598 Å, covering a range of 20 to 90 degrees in increments of 0.0392 degrees, with each step measured for 0.45 seconds. Raw data was collected and processed using Origin software and eventually the positions and intensities of the peaks were compared to those of the appropriate standards using the PDF database with PCPDF-WIN software provided by the JCPDS-International Centre for

Diffraction Data. The products of reduction roasting-magnetic separation process were also analysed through XRD for phase transformation observations. The as-received sample was also taken for SEM analyses.

A scanning electron microscope (SEM) is a device that produces images by scanning the surface of a sample with a concentrated beam of electrons. The interaction between electrons and atoms in the sample's surface layer produces signals that provide details about the surface's structure and composition(Choisez *et al.*, 2022; Hessels, 2023). SEM analysis provides high-resolution imaging of iron ore samples, revealing their mineralogical composition, which consists of iron-bearing minerals such as hematite and magnetite, along with gangue minerals such as quartz and clay, offering insight into the ore's appropriateness for reduction roasting. Moreover, SEM aids in evaluating the separation of iron minerals from gangue minerals and understanding their association, which is crucial for optimizing separation methods in iron ore beneficiation. JSM-7100F Scanning microscope shown in Fig. 15b operating at a 20 kV accelerating voltage and an oxford EDS was used to conduct SEM analyses.



Figure 15. a) D8 Advance Bunker XRD; b) JSM-7100F Scanning Electron Microscope

Post beneficiation techniques samples were taken for chemical, mineralogical and surface morphology analyses just as the initial raw sample for a comparative analysis to determine changes brought upon by the beneficiation process. The waste (non-magnetic tailings) was discarded as slurry post magnetic separation.

### **3.2 Process Optimization of Iron ore beneficiation by Taguchi and Experimental Validation.**

#### **3.2.1 *Experimental design***

The L9 orthogonal array, based on Taguchi method, was employed to carry out reduction roasting experiments at three different levels, involving three process factors: temperature, time, and coal dosage. The Taguchi experimental design is an efficient method that saves both time and money by minimizing the number of experiments needed to evaluate the impact of various factors during reduction roasting. In this study, the key response variables are iron grade and recovery which are determined through chemical and mineralogical analysis of magnetic concentrate obtained from roasted material through magnetic separation. To ensure accuracy, each experimental run was repeated, and the average results were used for statistical analysis. The primary goal of this statistical design is to identify the optimal factor values that will enhance both iron grade and recovery, and to determine the most significant factors in the process. As per the Taguchi method, 9 experiments are conducted, however due to duplication 18 runs were achieved in total before optimization of the reduction roasting process.

#### **3.2.2 *Reduction roasting and magnetic separation***

Reduction roasting entails transforming iron oxides like hematite and goethite into magnetite using carbonaceous reductants. Magnetite, being ferromagnetic, can then be separated from the gangue materials through low-intensity magnetic separation (Rayapudi, Agrawal and Dhawan, 2020). The roasting of iron ore was conducted in a laboratory Carbolite Gero (30-3000°C) muffle furnace (Fig. 17b) with the aid of high-quality refractory crucible. Transformation of hematite to magnetite is a complex process that involves several reactions as shown in equations: (10)(11)(12)(13)(14). A balanced stoichiometry reaction as shown in equation 3 was used to determine the

theoretical carbon requirement for complete hematite reduction. As a result, 50 g of low-grade iron ore was uniformly mixed with 16.6 g of non-coking coal. Two additional iron ore to coal ratios were determined by 15 and 20% excess stoichiometry and subsequently uniformly mixed. The prepared mixtures were thereafter roasted in a muffle furnace at different temperature ranges (700, 800 and 900°C) for 30, 60 and 90 minutes. In accordance with existing literature, this temperature range was selected as ideal for the conversion of hematite to magnetite (Sun *et al.*, 2014a; El-Husseiny *et al.*, 2015; Rayapudi, Agrawal and Dhawan, 2020). Tongs were used to remove the crucible post the roasting process, with the roasted sample allowed to cool before being ground as preparation for magnetic separation. The roasted samples were subjected to a drum magnetic separator (Fig. 16b) at 1800 gauss to discard non-magnetic tailings from the magnetic concentrate. Mass balance calculations were conducted to assess the efficiency of the reduction and separation processes. Initially, the mass of the low-grade iron ore was recorded before the reduction roasting, providing a baseline for comparison. After the roasting process, the mass was measured again to account for any weight loss due to moisture and volatiles. Subsequently, following magnetic separation, the masses of the magnetic concentrate and non-magnetic tailings were obtained. These measurements allowed for a comprehensive evaluation of the yields and losses throughout the processes, offering valuable insights into their overall effectiveness.



Figure 16. Equipment used in the beneficiation process; a) Carbolite Gero furnace; b) Drum magnetic separator

### 3.2.3 Statistical Analysis

The Taguchi design utilizes the signal-to-noise ratio (S/N) method to forecast variations in the performance of a response parameter. In this context, the signal refers to the target output value, while noise represents the unwanted value. This ratio is used to assess how much the quality feature diverges from the intended value. The ratio characteristics can be broadly categorized into three groups: larger the better; smaller the better; and nominal the better. The larger-the-better quality characteristic was employed in order to obtain maximum iron recovery and highest possible Fe grade. The Taguchi method employs orthogonal array designs to explore the entire parameter space with minimal experiments. This approach utilizes the signal-to-noise (S/N) ratio to assess the variability of each response across various parameter levels (Rath and Rao, 2017b).

### 3.2.4 Reaction kinetics modelling and thermodynamics experimental validation

#### *Reaction kinetics modelling*

The reduction of iron ore in this study follows a gas-solid reaction mechanism, where carbon monoxide ( $CO$ ) produced by the combustion of coal reduces hematite oxides. The reduction kinetic steps can be broken down into several key steps:

1. Production of carbon monoxide ( $CO$ )
2. Transportation of  $CO$  through the gas film to the ash interface.
3. Diffusion of  $CO$  through the ash layer to the reaction interface.
4. Adsorption and chemical reactions at the unreacted core surface.
5. Release of  $CO_2$  at the reaction interface.
6. Transportation of  $CO_2$  through the ash layer and gas film.

The experimental data were analysed to pinpoint the rate-controlling steps in the reduction process of iron oxides. The rate-limiting step was identified by assessing the linearity of the plots for both product layer diffusion control and chemical reaction control over time. To identify the rate-controlling step, the experimental data were fitted into two models:

$$1 - (1 - \alpha)^{\frac{1}{3}} = kt$$

*Chemical reaction control model*

$$1 - \frac{2}{3}\alpha(1 - \alpha)^{\frac{2}{3}} = kt$$

*Product layer diffusion control model*

Arrhenius equation was used to describe the temperature dependence of the reaction rate.

$$k = Ae^{-\frac{Ea}{RT}};$$

Whereby:  $k$  = rate constant,  $A$  = pre-exponential factor (frequency factor),  $Ea$  = activation energy (in J/mol),  $R$  = universal gas constant (8.314 J/mol·K),  $T$  = absolute temperature (in K).

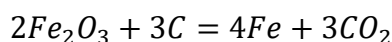
To determine the rate constants at different temperatures, the following equation was used.

$$\ln k = \ln A - \frac{Ea}{R} \cdot \frac{1}{T},$$

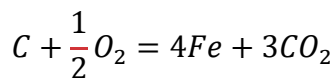
This logarithmic form of the Arrhenius equation allows for linear regression analysis to determine  $A$  and  $Ea$ .

### *Thermodynamics*

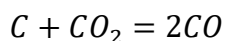
The thermodynamic analysis of the reduction roasting of iron ore with powdered non-coking coal as a reductant provides insights into the efficiency and feasibility of the process. The reduction of roasting of iron ore with powdered non-coking coal entails a series of chemical reactions. The main reactions can be summarized as follows:



#### *Reduction of hematite to metallic Fe*



#### *Oxidation of carbon-to-carbon monoxide*



#### *Boudouard reaction*

The thermodynamics of these reactions were examined using the concepts of chemical equilibrium and Gibbs free energy. Gibbs free energy ( $\Delta G$ ) is calculated using the formula.

$$\Delta G = \Delta H - T\Delta S,$$

Where  $\Delta H$  represents the change in enthalpy,  $\Delta S$  is the change in entropy, and  $T$  is the absolute temperature.

## CHAPTER 4

### RESULTS AND DISCUSSIONS

This chapter devoted to the results and discussion of low-grade iron ore and non-coking coal characterisation focusing on their potential utilization for iron ore beneficiation through reduction roasting and magnetic separation. An in-depth analysis of the beneficiation process results, kinetics and thermodynamics validation discussed

#### 4.1 Chemical analysis

The chemical analysis of the raw iron ore revealed that the sample consists of 56% *Fe*, 8.6% *Si*, 6,7% *Al* and 26,9 % oxygen. This composition is significant as it indicates that the ore has a relatively high iron content and can potentially be upgraded through reduction roasting and magnetic separation to meet the iron and steelmaking ore grade requirement (Faris *et al.*, 2017).

The presence of silica and alumina suggests potential challenges in the subsequent beneficiation process, as high levels of these impurities can affect the quality of the final iron product (Ghosh and Chatterjee, 2010). Although present, does not significantly detract from the ore's viability for beneficiation.

The proximate analysis of Morupule non-coking coal done by TGA presented in Table 7 establishes that the coal consists of  $\approx 50\%$  fixed carbon which is a critical factor for its effectiveness as a reductant in the reduction roasting process. Therefore, this validated the use of this local coal as a reductant in the iron ore concentration process since non-coking coal(s) with similar or less carbon content has been previously used in successful iron ore reduction studies.

Table 7. Coal proximate analysis on a wet basis by weight

| Fixed Carbon<br>(FC) % | Ash Content, % | Moisture Content, % | Volatile<br>matter (VM) | Total |
|------------------------|----------------|---------------------|-------------------------|-------|
| 50.01                  | 21.13          | 4.35                | 24.51                   | 100   |

(Ravisankar, Venugopal and Bhat, 2019) conducted an effective study on coal-based reduction roasting and the subsequent magnetic separation of iron ore utilizing semi-bituminous coal with a fixed carbon content of 48.83%. A product with a 66.6% iron content was obtained, achieving a 90.4% recovery rate from the original iron ore, which had an iron content of 58%. In another research work regarding low grade iron ore enrichment (47.1% Fe) (Roy, Nayak and Rath, 2020a), coal fines containing 52.6% fixed carbon were used as a reductant in the reduction roasting and magnetic separation process, leading to an increase in Fe grade to 58.52% and an overall Fe recovery of about 89.63%. The ultimate analysis conducted on the Morupule coal samples confirmed its classification as non-coking coal. This analysis provided essential data on the coal's elemental composition, including carbon, hydrogen, sulphur, and ash content. These components' quantities are furnished in Table 8.

The results indicated that the coal possesses a low H/C and O/C ratios in non-coking coal, along with its higher carbon content making it suitable for reduction processes where efficient carbon sources are required. It suggests that non-coking coal can provide a more stable carbon source during reactions due to a lower H/C ratio indicating a higher carbon content relative to hydrogen and an O/C ratio of approximately 0.174 (9.77% O and 56.10% C), this suggests that the coal is relatively low in oxygen, making it more suitable for reduction processes as it can yield more carbon monoxide (CO) when combusted.

For non-coking coal to be considered effective as a reductant in metallurgical applications, it should ideally have carbon content: 50 - 80%, volatile matter: 20 - 40% and ash content below 10%(Carpenter, 2004; Nezhad, Hemmati and Vakili-Azghandi, 2022). Thus, the characterization of the coal supports its utilization in the reduction roasting of iron ore.

Table 8. Coal ultimate analysis on a wet basis by weight

| Element          | C     | H    | N    | S    | O     | Ash<br>content | Moisture<br>content | Total |
|------------------|-------|------|------|------|-------|----------------|---------------------|-------|
| Abundance<br>(%) | 52.10 | 2.32 | 1.20 | 0.53 | 18.37 | 21.13          | 4.35                | 100   |

## 4.2 X-Ray Diffraction studies

The XRD diffraction pattern of the sample, illustrated in Fig. 17, reveals that the ore contains mineral phases including hematite, magnetite, and silica (quartz). The pattern further indicates that hematite is the predominant phase in the raw iron ore. The presence of hematite is significant, as it is a primary source of iron and is amenable to reduction processes in the transformation of hematite to magnetite for subsequent magnetic separation. Prior studies have shown that ores with high hematite content yield better recovery rates when subjected to reduction roasting, corroborating our findings of predominant hematite in our sample (Liu *et al.*, 2014; Kukkala *et al.*, 2024).

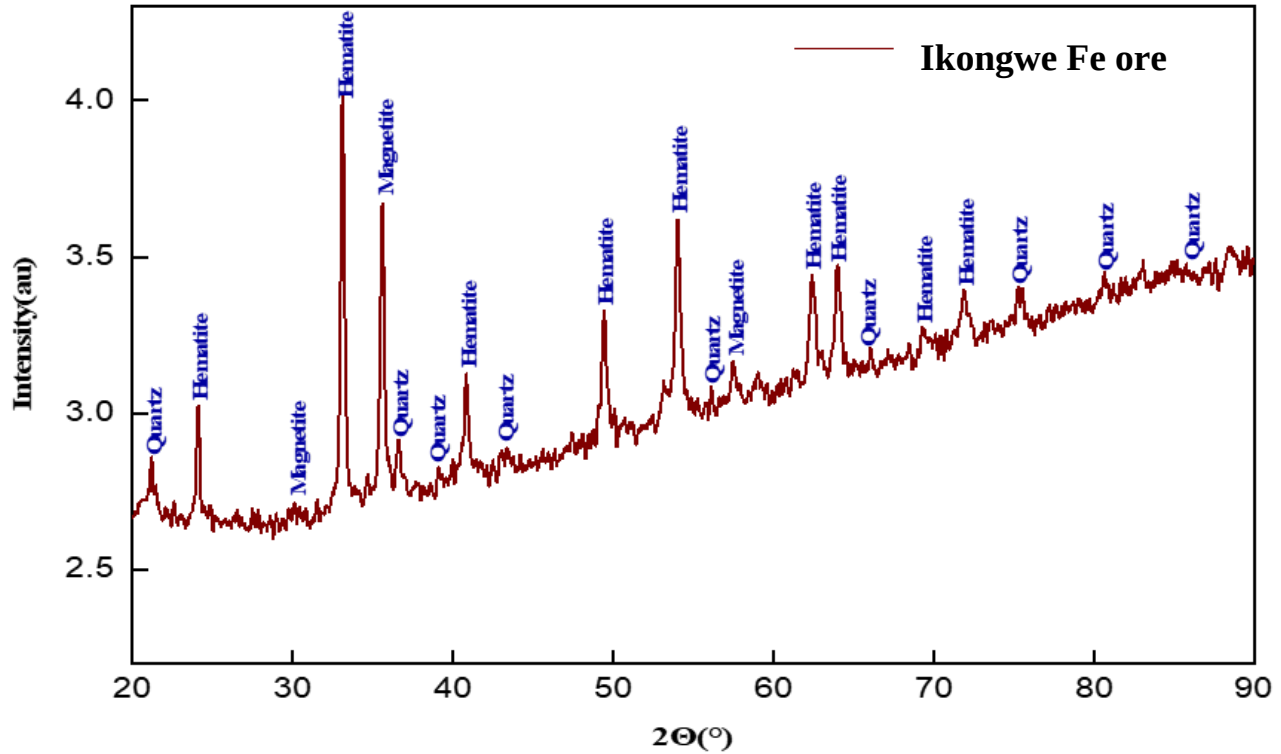


Figure 17. X-ray diffraction for raw iron ore sample

The ore also contains partial compositions of magnetite and quartz. Magnetite can be more stable and may require higher temperatures or longer processing times to reduce fully to iron. Thus, having less magnetite can simplify the reduction roasting process. The low gangue content of 27.1% enhances the reduction process by increasing iron oxide yield, reducing energy costs, and minimizing slag production, leading to a more efficient and sustainable operation.

A quantitative XRD analysis was performed by measuring the area of each peak corresponding to a specific phase relative to the total area under all peaks. The results are provided in Table 8.

Table 8. Raw iron ore quantitative X-ray diffraction analysis.

| Mineral   | Formula   | Mineral abundance (%) |
|-----------|-----------|-----------------------|
| Hematite  | $Fe_2O_3$ | 54.6                  |
| Magnetite | $Fe_3O_4$ | 18.3                  |

|           |           |        |
|-----------|-----------|--------|
| Silica    | $SiO_2$   | 13.8   |
| Alumina   | $Al_2O_3$ | 5.5    |
| Amphibole |           | 7.8    |
| Total     |           | 100.00 |

### 4.3 Scanning Electron Microscopy (SEM) and Energy Dispersive spectroscopy (EDS) analysis

The surface morphology of the raw iron ore sample was analysed using scanning electron microscopy (SEM). The SEM images, as shown in Fig. 18, reveal that the unroasted iron ore particles exhibit an angular and irregular morphology, which is characteristic of hematite. The angular and irregular shapes of hematite particles can enhance the surface area available for reaction during reduction, potentially leading to improved iron recovery rates.

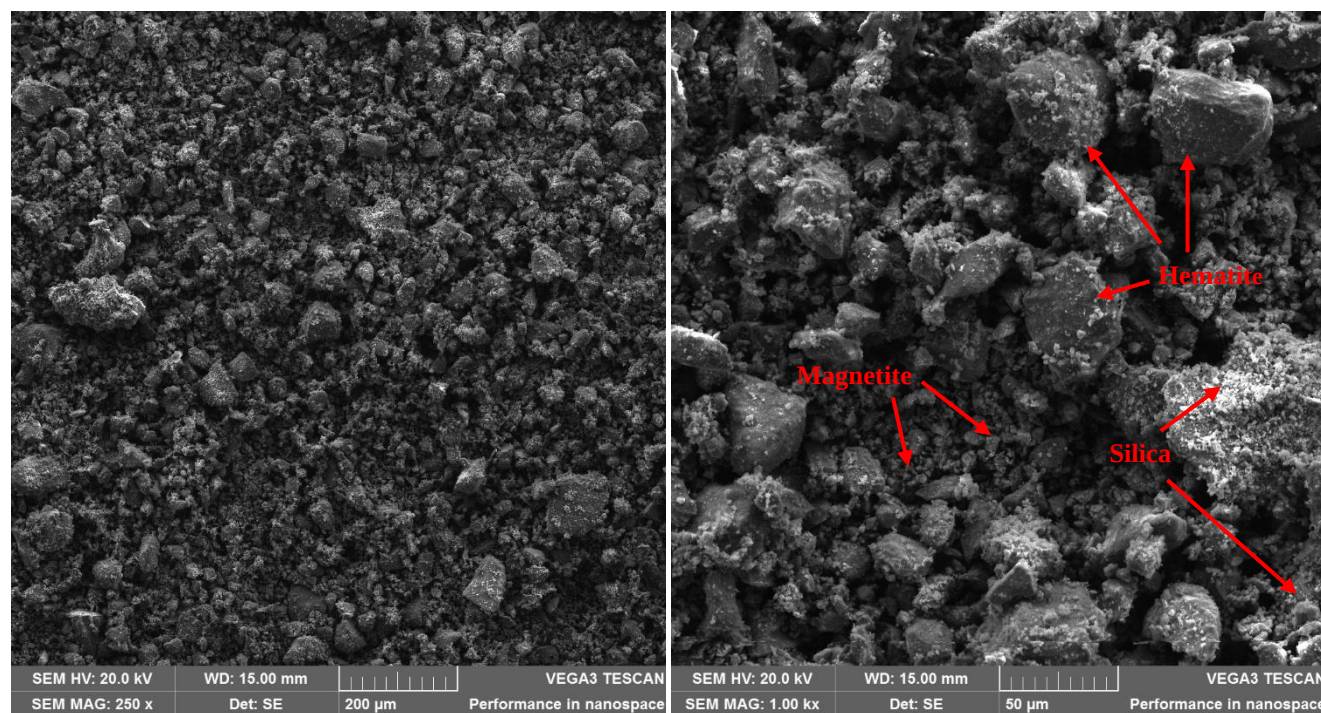


Figure 18. Secondary Electron images of raw iron ore

Studies have shown that ores with angular hematite particles often exhibit better reactivity during reduction processes, which supports our findings regarding the morphology of the iron ore sample (Zhao *et al.*, 2021; Poojari, Vardhan and Jathanna, 2023). In contrast, traces of magnetite are indicated by more regular, spherical shapes.

This morphology is significant as it suggests the presence of different iron mineral phases, which can influence the behaviour during reduction roasting and subsequent magnetic separation. The shades of grey observed in the SEM images correlate with differences in material composition and density, with lighter grey areas suggesting the presence of impurities such as silica and alumina. The SEM micrograph of the feed material also reveals that hematite is distributed within a matrix of silica and clay.

The energy dispersive spectroscopy (EDS) shown in Fig. 19 provides an estimate of the feed material primary elemental composition. The EDS mapping indicates that the feed is predominantly made up of iron, silicon, aluminium and oxygen. Table 9 furnishes primary elemental composition, and their abundances as determined by EDS. The high iron content confirms the suitability of the ore for reduction processes, while the presence of silicon and aluminium highlights the gangue materials that beneficiation process should eliminate.

*Table 9. EDS elemental composition*

| <b>Constituent</b> | <b>Fe</b> | <b>Si</b> | <b>Al</b> | <b>O</b> | <b>Others</b> |
|--------------------|-----------|-----------|-----------|----------|---------------|
| <b>Wt. %</b>       | 56.1      | 8.6       | 6.7       | 26.9     | 1.7           |

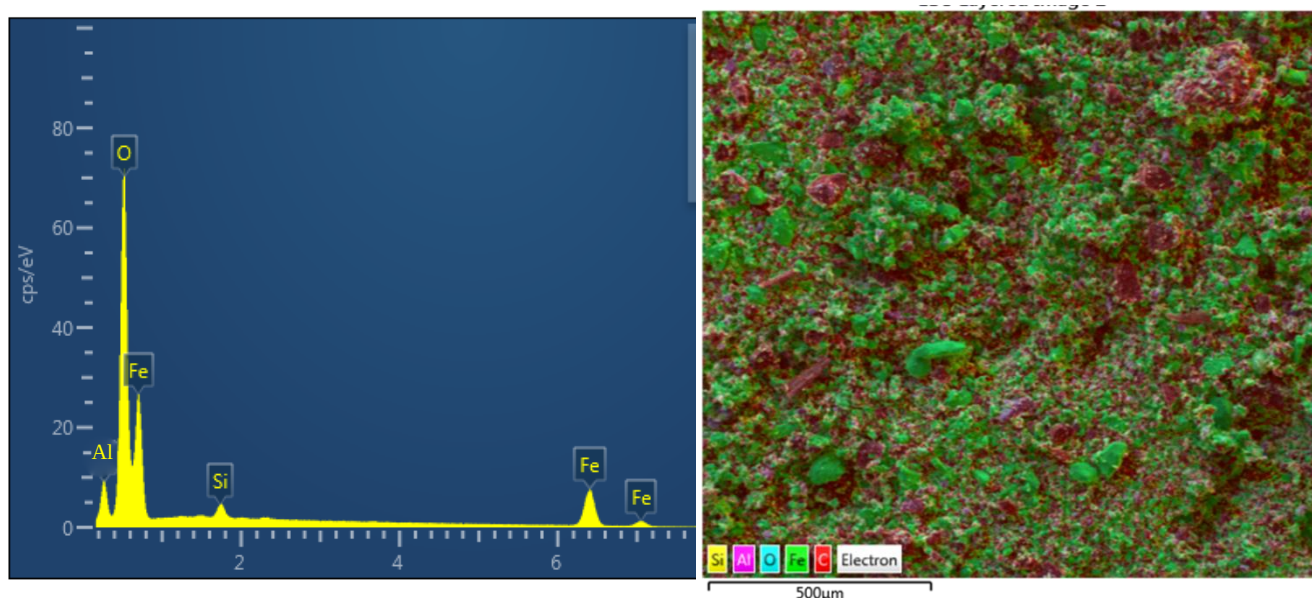


Figure 19. a) Raw iron ore EDS spectrum; b) EDS mapping

The EDS estimate (56% Fe, 2.9% Si, 3.7% Al, and 23.5% O) aligns with the elemental data provided by XRF. EDS is typically more surface-sensitive, while XRF analyses the bulk composition, and their correlation confirms that the feed material's surface composition is representative of its bulk. The elemental data from EDS helps in interpreting the phases detected in XRD, as the elements (iron, silicon, aluminium, and oxygen) likely form specific oxide compounds that XRD can detect.

## 4.4 Beneficiation studies

### 4.4.1 Mass Balance Analysis

Understanding the mass balance through experimental runs provides essential insights into the efficiency and dynamics of this process. The experimental data in Table 10 presents variations in initial mass, with weights ranging from 27.0 g to 34.0 g. The variability in initial mass reflects different iron ore to coal ratios, which can influence both reduction efficiency and weight losses. The run with an initial mass of 27.0 g corresponds to a ratio of 3:1 (iron ore to coal), while the

other runs, with masses 30.5 g to 34.0 g, containing 20% and 30% excess coal based on stoichiometric calculations.

*Table 10. Reduction roasting and magnetic separation mass balances*

| <b>Run</b> | <b>Initial mass(g)</b> | <b>Mass after roasting (g)</b> | <b>Roasting weight loss (g)</b> | <b>Mass after Magnetic separation (g)</b> | <b>Overall weight loss (g)</b> |
|------------|------------------------|--------------------------------|---------------------------------|-------------------------------------------|--------------------------------|
| 1          | 34.0                   | 26.6                           | 7.4                             | 18.5                                      | 15.5                           |
| 2          | 27.0                   | 24.3                           | 2.7                             | 17..2                                     | 9.8                            |
| 3          | 30.5                   | 27.6                           | 2.9                             | 17.3                                      | 13.2                           |
| 4          | 30.5                   | 26.8                           | 3.7                             | 20.2                                      | 10.3                           |
| 5          | 34.0                   | 27.9                           | 6.1                             | 20.6                                      | 13.4                           |
| 6          | 27.0                   | 22.4                           | 4.6                             | 17.6                                      | 9.4                            |
| 7          | 34.0                   | 27.4                           | 6.6                             | 18.9                                      | 15.1                           |
| 8          | 27.0                   | 21.9                           | 5.1                             | 15                                        | 12                             |
| 9          | 30.5                   | 26.4                           | 4.1                             | 14.8                                      | 15.7                           |

### *Temperature Effects*

- 700°C (Runs 1-3): Lower roasting temperatures resulted in varied weight losses, with Run 2 showing the least loss, indicating potentially effective reduction at the lower iron ore to coal ratio (3:1).
- 800°C (Runs 4-6): Moderate temperatures showed increased weight losses, particularly in Run 5 (6.1 g), suggesting greater volatility or inefficiencies with excess coal.
- 900°C (Runs 7-9): Higher temperatures yielded significant weight losses but also varied mass after magnetic separation. Notably, run 9 experienced a high overall weight loss of 15.7 g, highlighting potential losses associated with high temperatures and excess coal.

### *Iron Ore to Coal Ratios*

- 3:1 Ratio: Present in Run 2 (27.0 g), which exhibited relatively low roasting weight loss and reasonable post-separation mass.
- 20% and 30% Excess Coal: Other runs with excess coal demonstrated higher weight losses, indicating potential inefficiencies or loss of valuable material during the process. For instance, run 1 with 34.0 g and 7.4 g weight loss suggests the presence of unreacted coal or volatiles.

The mass balance analysis of iron ore roasting provides valuable insights into the reduction process; however, it must be supplemented with chemical and mineralogical analyses to accurately determine iron yield. Understanding the relationship between mass changes and actual iron recovery is essential for optimizing the roasting process.

#### **4.4.2** *Experimental design*

In this research, an L9 orthogonal array experimental design, following the Taguchi method, was employed to investigate the effects of three process parameters on the reduction roasting of iron ore. The process took place in a muffle furnace under controlled atmospheric conditions and was subsequently subjected to magnetic separation using a low-intensity drum separator set at a magnetic field strength of 1800 gauss. The three parameters examined, and their respective levels are as follows:

- i. Temperature: 700, 800, 900 °C
- ii. Time: 30, 60, 90 minutes.
- iii. Coal dosage: 25, 40, 50 % (by weight of iron ore)

The Taguchi L9 experimental design matrix and response variables is shown in Table 11. This design allowed for the systematic evaluation of effects and potential interactions between the selected process parameters on the iron ore beneficiation performance.

Table 11. Taguchi L9 experimental conditions and corresponding response variables

| Exp. No | Experimental conditions |           |             | Response variables |             |
|---------|-------------------------|-----------|-------------|--------------------|-------------|
|         | Temperature, ° C        | Time, Min | Coal dosage | Grade, %           | Recovery, % |
| 1       | 700                     | 30        | 25          | 63.7               | 78.1        |
| 2       | 700                     | 60        | 40          | 64.3               | 77.5        |
| 3       | 700                     | 90        | 50          | 65.1               | 56.1        |
| 4       | 800                     | 30        | 40          | 68.5               | 82.5        |
| 5       | 800                     | 60        | 50          | 67.4               | 70.6        |
| 6       | 800                     | 90        | 25          | 65.7               | 72.6        |
| 7       | 900                     | 30        | 50          | 64.3               | 60.8        |
| 8       | 900                     | 60        | 25          | 62.5               | 54.9        |
| 9       | 900                     | 90        | 40          | 63.2               | 49.7        |

The iron grade was determined by the means of XRF technique whereas the recovery was calculated using Equation 15.

$$R = \frac{C \times c}{F \times f} \times 100$$

( 15 )

*R is the Fe recovery (%), C and c are the magnetic product weight and grade (%), respectively, whereas F is the feed sample weight and f are the feed sample grade.*

Ultimately the magnetic product grade and recovery was analysed and measured to evaluate the effectiveness and optimization of the beneficiation process. The results obtained from the Taguchi based design were further analysed to establish optimal roasting values and the relative effects of process parameters in the beneficiation process. This information was crucial in understanding the underlying mechanisms and developing a robust iron ore beneficiation process.

#### 4.4.3 Extent of reduction roasting degree

In this research, the reduction kinetics of the roasted iron ore samples, following the Taguchi L9 design, were analysed in terms of the relationship between the level of hematite reduction and the duration of reduction. The degree of reduction ( $\alpha$ ) was determined using the following equation:

(4). Table 12 presents the degree of reduction, defined as the percentage of iron oxide transformed into metallic iron (Fe) after the reduction roasting process.

Calculation for determination of reduction degree for Experiment 1:

$$\alpha = \frac{WLt}{WL} = \frac{m_0 - m_t}{m_0 + m_c + m_v} = \frac{27.00 - 19.53}{6.10 + 3.40 + 1.70} = \frac{3.43}{11.20} = 0.30625g \times 100\% = \sim 30.6\%$$

Similarly, calculations for other experiments 2-9 were calculated.

The fractional loss of oxygen from hematite ( $\alpha$ ) during the reduction roasting of iron ore is evaluated for the isothermal reduction kinetics study by altering the reduction temperature and duration. It is defined as follows:  $= \frac{Mt}{Mo}$ ; Where,  $Mt$  is the oxygen weight loss from hematite ( $Fe_2O_3$ ) for a determined time (t, minutes) and  $Mo$  denotes the total hematite oxygen. Fractional oxygen losses of the experimental runs are provided in Table 10. Therefore,  $\alpha$  Vs time with temperature variations plot is furnished in Fig. 20.

Table 12. Taguchi-based experimental runs reduction degrees and fractional oxygen loss

| Experiment | Temperature, (° C) | Time, (min) | Coal dosage, (%) | Reduction degree, (%) | Oxygen loss, (%) |
|------------|--------------------|-------------|------------------|-----------------------|------------------|
| 1          | 700                | 30          | 25               | 30.6                  | 16.9             |
| 2          | 700                | 60          | 40               | 28.8                  | 23.2             |
| 3          | 700                | 90          | 50               | 35.8                  | 37.6             |
| 4          | 800                | 30          | 40               | 25.2                  | 20.2             |
| 5          | 800                | 60          | 50               | 34.1                  | 35.9             |
| 6          | 800                | 90          | 25               | 41.1                  | 22.7             |

|   |     |    |    |      |      |
|---|-----|----|----|------|------|
| 7 | 900 | 30 | 50 | 36.9 | 38.8 |
| 8 | 900 | 60 | 25 | 45   | 24.9 |
| 9 | 900 | 90 | 40 | 48.3 | 38.8 |

The effects of residence time over different temperatures on the reduction degree were analysed using a main effects plot, as shown in Fig. 20. The plot indicates that,

- Temperature has a major positive impact on the degree of reduction. The reduction degree increased significantly from 700°C to 800°C, with a peak at 800°C, followed by a slight decline at 900°C. Similar trends are observed in the literature, where 800°C is often reported as an optimal temperature for reduction roasting of iron oxides, particularly hematite, with a slight decline in reduction efficiency at higher temperatures due to sintering or re-oxidation. (Yu *et al.*, 2017; Roy, Nayak and Rath, 2020b) reported observed optimal reduction at 800°C, with diminished returns at higher temperatures. Increasing the temperature beyond 850°C resulted in a decrease in reduction efficiency due to sintering effects, which hinder gas-solid reactions.
- Time positively influenced the reduction degree up to 60 minutes, after which a decline was observed at 90 minutes. This behaviour is often explained by the balance between reaction kinetics and the sintering of iron particles, which can reduce the surface area available for further reduction. (Kukkala *et al.*, 2024) reported a similar trend was noted, where the best reduction degree was achieved between 45-60 minutes of roasting at temperatures between 700°C and 800°C. Prolonged residence times led to reduced surface area and, consequently, a lower reduction degree due to sintering. In another study by (Rath and Rao, 2017), the reduction of hematite at 700°C and 800°C showed optimal results around 60 minutes, with the reduction degree declining after 90 minutes, corroborating these findings.

- The coal dosage had a significant effect, especially at 900°C, where increasing the dosage from 25% to 50% substantially improved the reduction degree. This is consistent with findings in literature where an increase in reductant dosage enhances the reduction of iron oxides by providing more carbon to react with the oxygen in the oxides. (Sun *et al.*, 2014b; Faris *et al.*, 2017) observed that increasing the coal dosage in reduction roasting experiments led to higher degrees of reduction due to the greater availability of carbon, which enhances the formation of reducing gases like  $CO$  and  $CO_2$ .

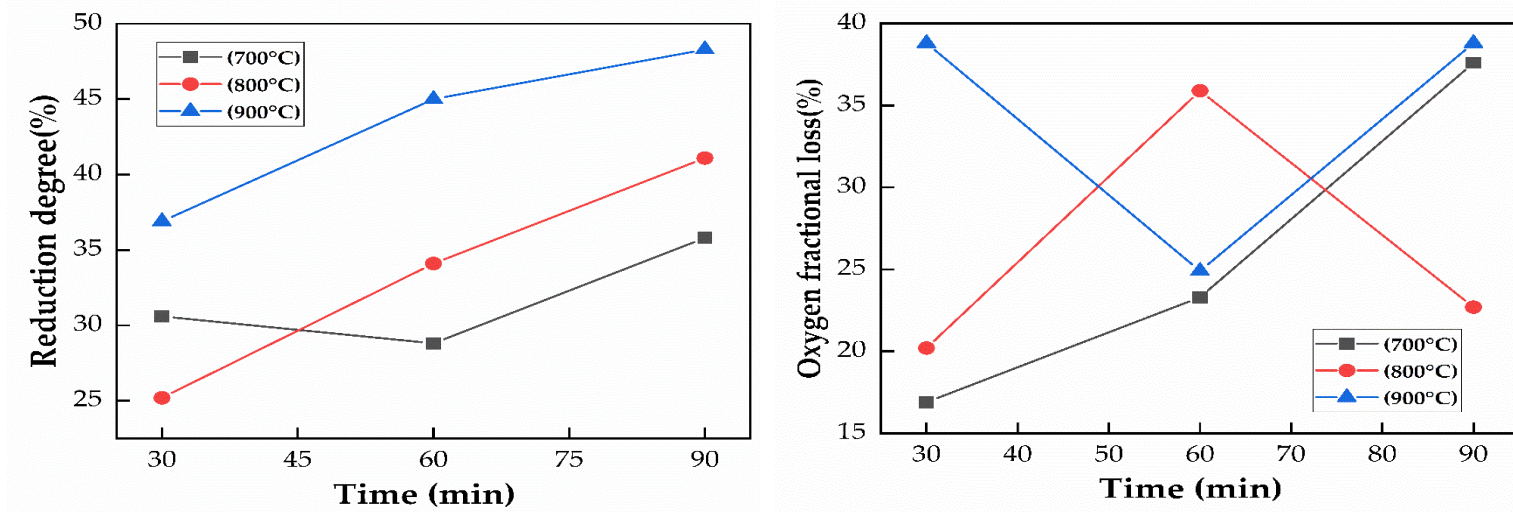


Figure 20. (a) Reduction degree versus time at different temperature, (b) Oxygen fractional loss versus time

The coal dosage not only provides the carbon necessary for reducing iron oxides but also affects the oxygen removal process. Increasing the coal dosage leads to more complete reduction, as evidenced by the higher oxygen loss (e.g., 38.8% in experiments 7 and 9). This is supported by the elemental analysis from EDS and XRF, which shows the removal of oxygen from iron oxides.

## 4.5 Optimization

### 4.5.1 Chemical analysis

The initial chemical analysis of the raw iron ore showed that the sample contained 56% Fe, 5.3% Si, 4.9% Al and 10.8% oxygen. These values indicated a moderate iron content and relatively high levels of silica and alumina, which are common impurities that negatively impact the iron ore beneficiation process. The primary objective of this study was to upgrade the iron content of the ore to meet the iron and steelmaking grade requirements through reduction roasting and magnetic separation.

After subjecting the ore to reduction roasting at 800°C for 60 minutes with a coal dosage of 50%, the iron content increased to 68.2%, demonstrating a significant improvement in the iron concentration. This increase in Fe content, as shown in Table 13, validates the effectiveness of the reduction roasting process, which successfully transformed non-magnetic iron oxides (such as hematite) into magnetite, enabling the subsequent magnetic separation to enrich the iron content.

Table 13. Comparison between raw and roasted ores chemical analysis

| Constituent                    | As-received Mass (%) | Roasted Mass (%) |
|--------------------------------|----------------------|------------------|
| Fe                             | 56                   | 68.2             |
| SiO <sub>2</sub>               | 13.5                 | 6.8              |
| Al <sub>2</sub> O <sub>3</sub> | 5.5                  | 2.8              |

In addition to the iron content improvement, the reduction roasting process led to a decrease in the masses of impurities. The SiO<sub>2</sub> content was reduced from 13.5% to 6.8%, and the Al<sub>2</sub>O<sub>3</sub> content decreased from 5.5% to 2.8% in the roasted ore. The reduction in these impurities is critical, as both silica and alumina are known to negatively affect the quality of iron ore concentrates by increasing slag formation during smelting, thus increasing the energy consumption and decreasing the yield of usable iron.

The results from this study are consistent with findings in existing literature, where reduction roasting followed by magnetic separation is widely recognized as an effective beneficiation method for low-to-medium grade iron ores. Studies by (Muriana, Muzenda and Abubakre, 2014; Rayapudi, Agrawal and Dhawan, 2020) demonstrated similar trends in iron content enhancement and impurity reduction through reduction roasting of iron oxides using carbon-based reductants.

The increase in Fe content to 68.2% observed in this study is comparable to the improvements reported in these studies, which typically show iron content improvements ranging from 65% to 70% after reduction roasting.

#### **4.5.2 Mineralogical and morphological studies of the roasted product**

##### *XRD studies*

The magnetic concentrate achieved from the reduction roasting at optimal conditions (800°C for 60 minutes with 0.5% coal dosage) was analysed using XRD to identify and confirm the newly formed phases and substantiate hematite to magnetite transformation. The XRD analysis of the magnetic concentrate (Fig. 21) shows a significant transformation with a significant increase in the magnetite phase to 65.3% from an initial 18.3% with hematite abundance also decreasing from 54.6 to 16%. This highlights the successful transformation of hematite-rich iron ore into a magnetite-dominant concentrate through controlled reduction roasting, as per the primary objective of this study and also consistent with existing literature, where similar conditions have resulted in successful phase transformations (Anderson *et al.*, 2014; Choisez *et al.*, 2022).

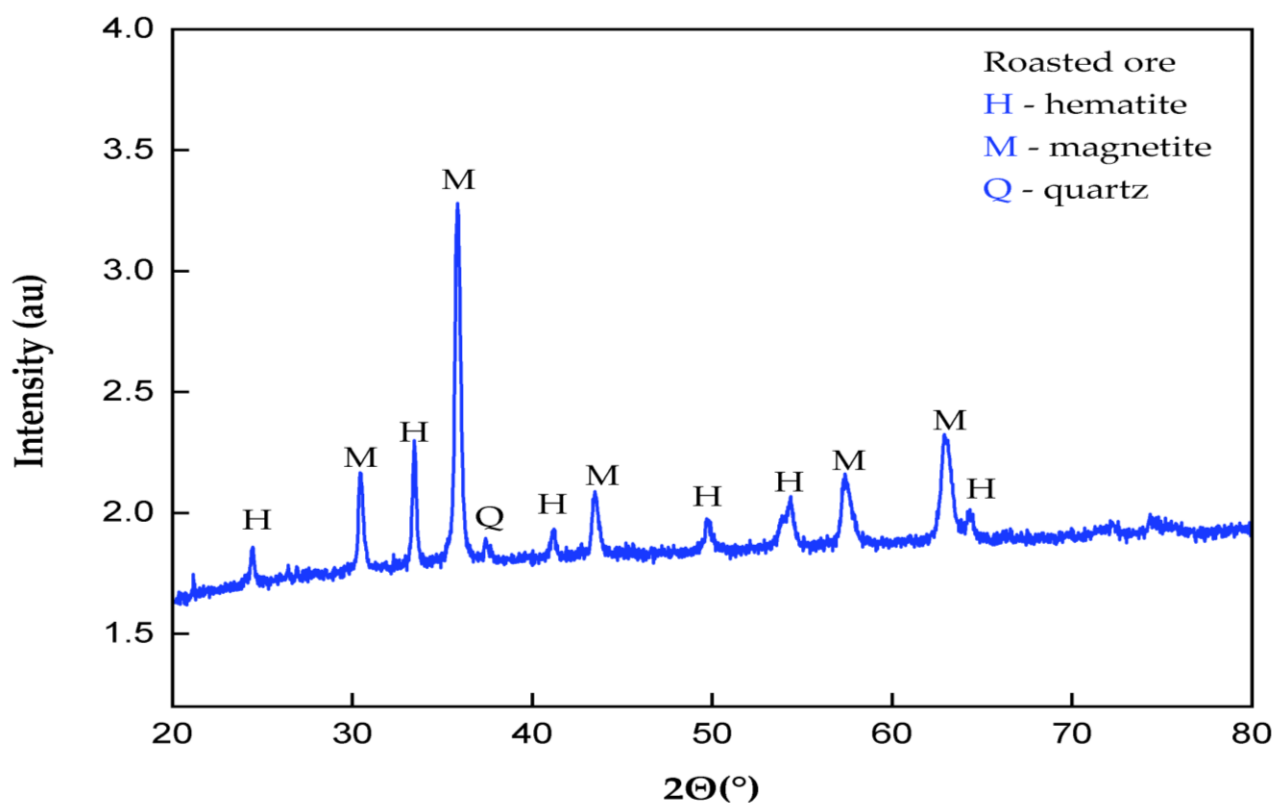


Figure 21. Optimized magnetic concentrate XRD diffractogram

Despite the successful transformation, traces of hematite (23.6%) remain in the magnetic fraction, indicating that the reduction was not fully complete. Incomplete reduction is a common occurrence in reduction roasting processes due to factors such as insufficient residence time, suboptimal reducing agent dosage, or the intrinsic properties of the ore itself. The presence of quartz in the magnetic fraction results from its entrapment and partial release from the iron phases.

The magnetic fraction also contains a modest amount of quartz (3%), which is likely due to the entrapment and partial release of these gangue minerals from the iron phases during the roasting and magnetic separation steps. Fig. 22 presents a comparison chart of mineral phases present in both the raw and roasted iron ores.

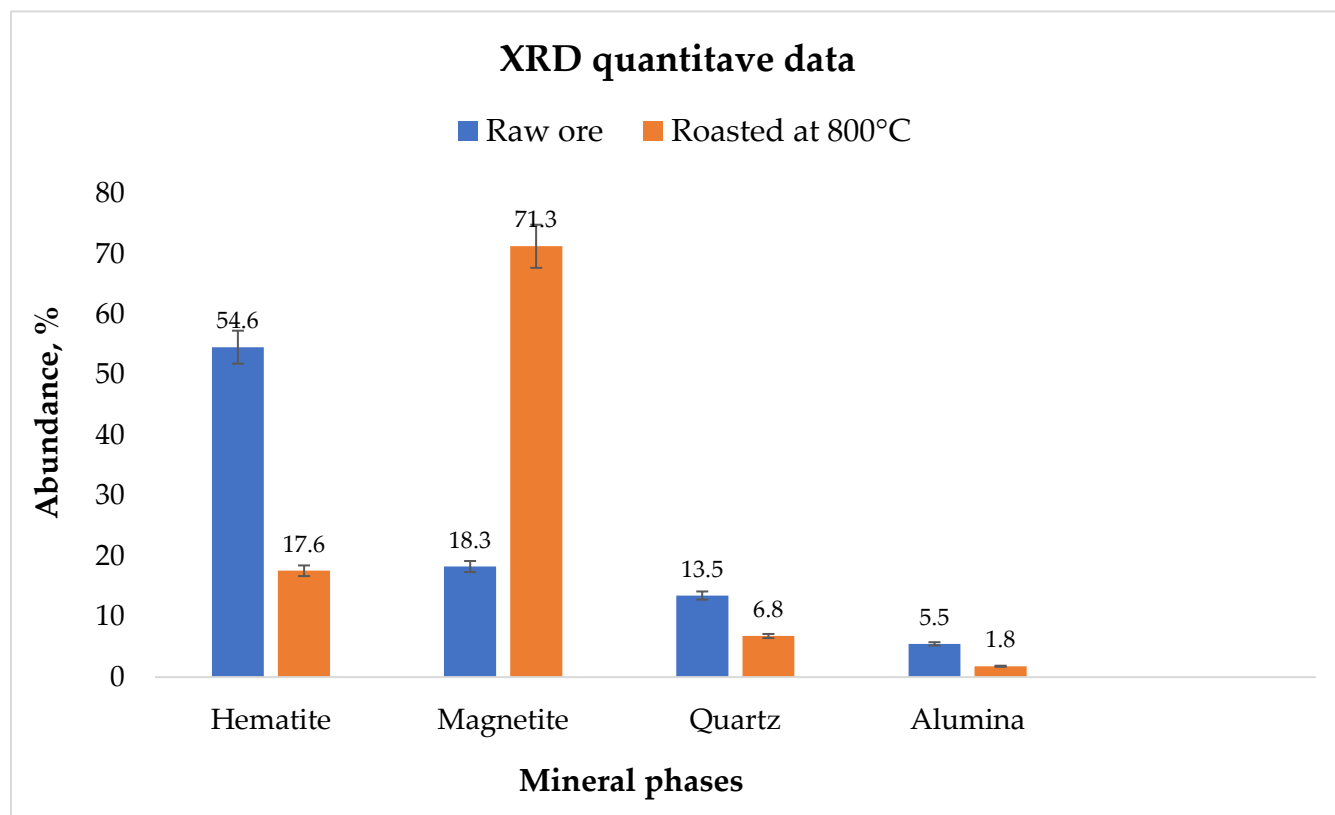


Figure 22. XRD quantitative data comparison bar chart

#### SEM-EDS studies

SEM was utilized to analyse the surface morphology and mineralogical alterations in the roasted product. The SEM images of the raw iron ore (a) and the subsequent magnetic concentrate (b)

reveal distinct morphological features that correlate with the reduction process as illustrated in Fig.23.

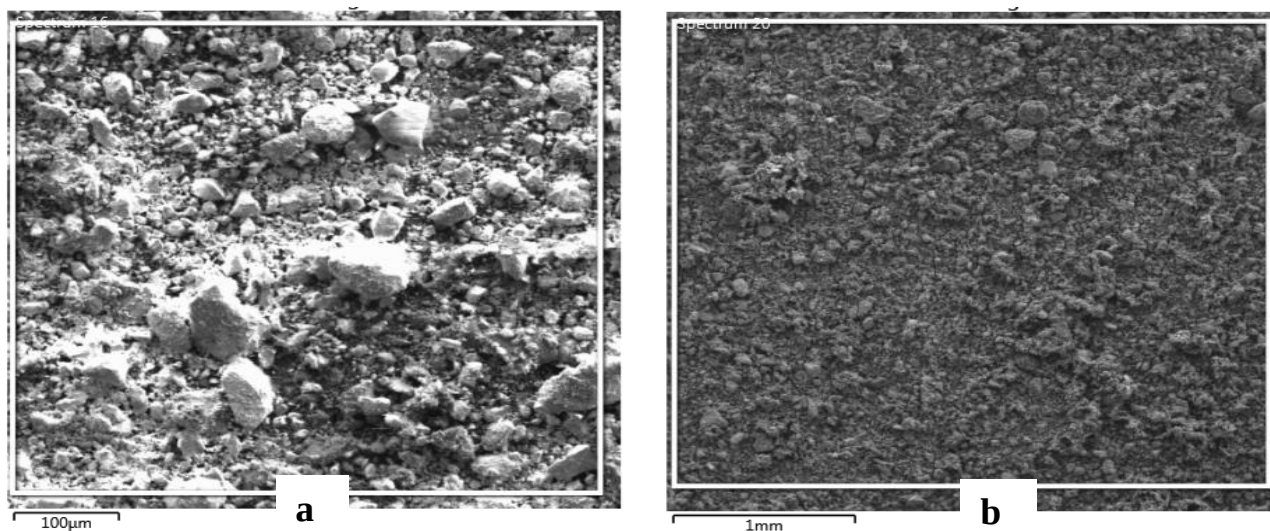


*Figure 23. Showing electron images of a) raw iron ore and b) magnetic concentrate*

The variation in shape and size of particles observed in the SEM images can be attributed to the changes in mineral structure as hematite is reduced to magnetite. The particle shape of hematite is usually more angular and uneven, while magnetite tends to form more rounded and uniform particles due to the recrystallization that occurs during the reduction process, as iron oxides are thermally altered and restructured.

The SEM images further show a transition from lighter to darker shades, indicating the successful reduction of hematite to magnetite and the removal of significant portions of gangue materials such as silica and alumina. The SEM analysis shows that the high-grade concentrate (upgraded from 56% Fe to 68% Fe) exhibits a more homogeneous distribution of magnetite. Images obtained from SEM (Fig. 24) also provide further insights into the microstructural changes that have occurred during the reduction process. The images reveal spongy textures and fractured mineral grains, particularly in iron-rich regions of the sample. These textural changes are indicative of the

conversion of hematite to magnetite, as the reduction process often leads to the formation of porous and fractured structures due to the release of oxygen from hematite.

The findings from the SEM analysis are also consistent with existing literature, where similar morphological transformations have been reported during the reduction of hematite to magnetite. Studies by ((Pedersen, Postma and Jakobsen, 2006; Rzepa *et al.*, 2016) have documented the recrystallization of iron oxide minerals during thermal reduction, leading to more uniform and magnetically susceptible particles.

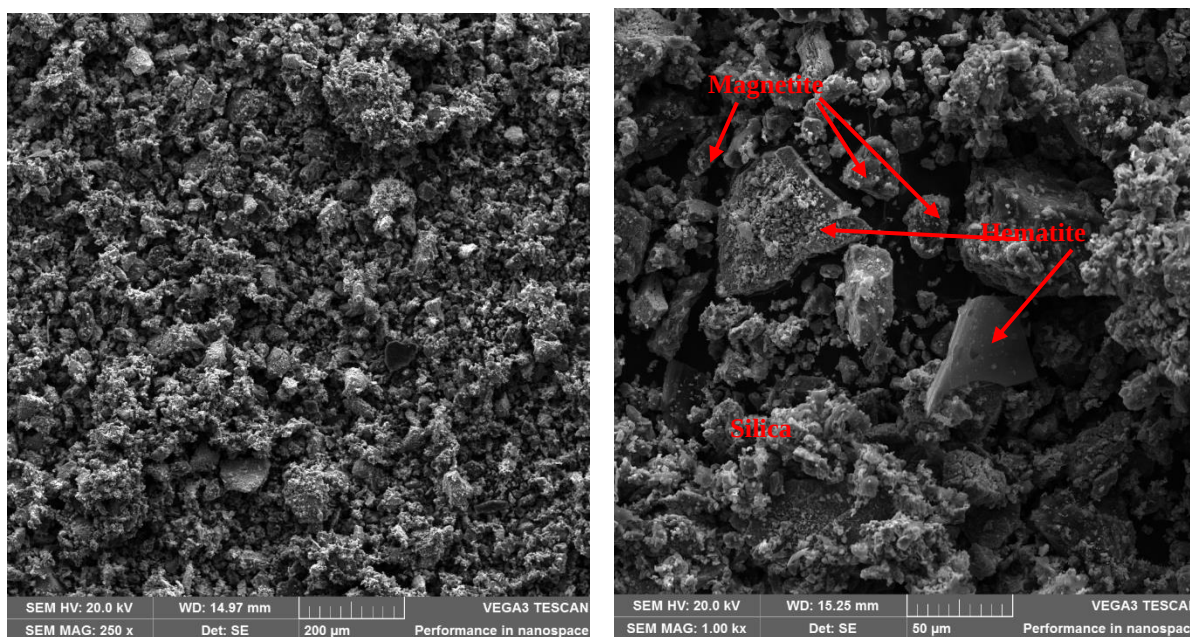


*Figure 24. Roasted ore images obtained from SEM*

The EDS analysis provides a detailed breakdown of the elemental composition of both the raw and roasted iron ore samples. The raw sample predominantly comprises iron (Fe), silicon (Si), aluminium (Al), and oxygen (O), as indicated by the mapping results (Figure 25 and Table 14). The high iron content confirms the suitability of the ore for reduction processes, while the presence of silicon and aluminium highlights the gangue materials associated with sample.

In the roasted product, the EDS results show a significant increase in iron content from 58 % in raw ore to 68 %, aligning with the observed morphological changes. The reduction process appears to have effectively removed a substantial amount of gangue, contributing to the higher purity of the magnetite concentrate. The elemental mass of aluminium and silicon decreased as shown in table 14. The elemental mass of oxygen reduces from 26.9 % to 10.8% in roasted sample attributed by oxygen requiring chemical reactions during roasting. Fig. 25 provides EDS spectrum and elemental mapping roasted iron ore showing concentration more concentration of iron across the sample.

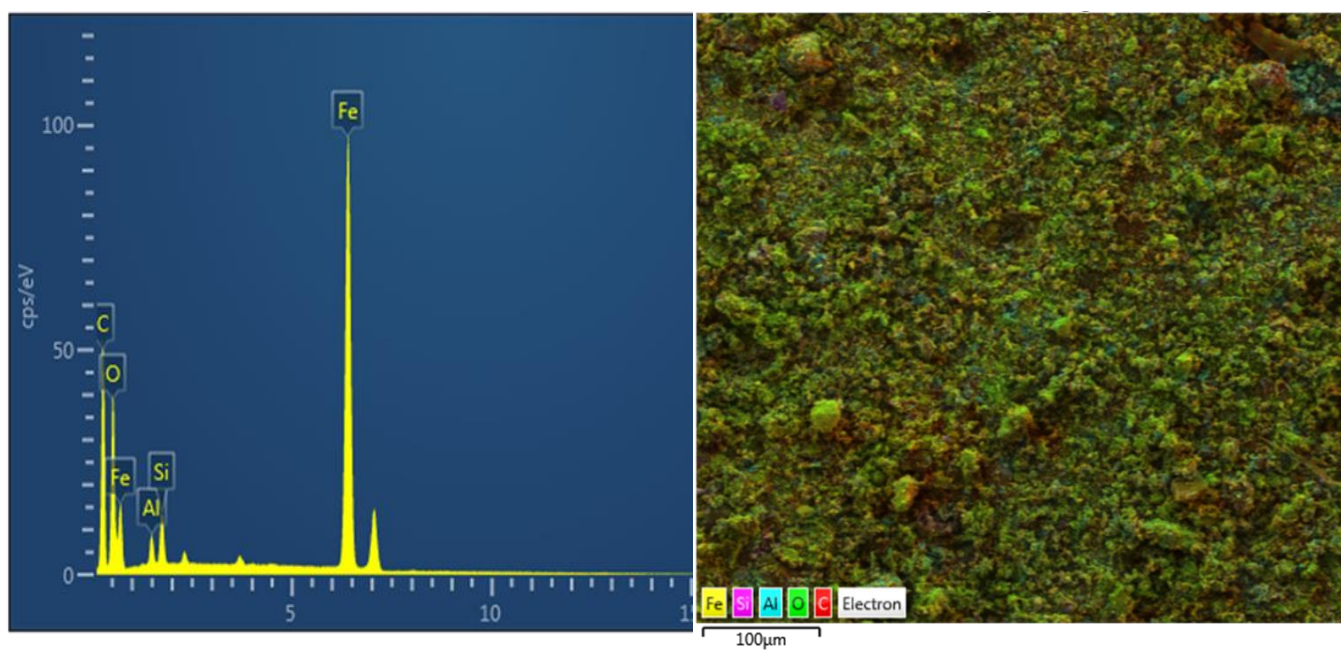


*Figure 25. Roasted iron sample EDS spectrum and elemental mapping*

The EDS data underscores the effectiveness of the reduction roasting and magnetic separation processes in enhancing the quality of the iron product. Table 14 furnishes a comparison of EDS elemental analysis between feed sample and the roasted sample obtained from optimum temperature of 800 °C.

Table 14. Elemental analysis of as-recieved and roasted at 800°C

| Elements  | As-received Mass (%) | Roasted Mass (%) |
|-----------|----------------------|------------------|
| Iron      | 56.1                 | 68               |
| Silicon   | 8.6                  | 5.3              |
| Aluminium | 6.7                  | 4.9              |
| Oxygen    | 26.9                 | 10.8             |
| Carbon    | -                    | 9.7              |
| Others    | 1.7                  | 1,3              |
|           | 100                  | 100              |

### Effect of roasting temperature

The study of the impact of roasting temperature on the reduction of iron ore shows a significant correlation between temperature and the response variables of grade and recovery as shown in Fig. 26. At a roasting temperature of 700 °C, the grade was measured at 64.2% Fe, with a recovery of 70.3%. Raising the temperature to 800 °C resulted in a notable enhancement, reaching a grade of 68.2% Fe and a recovery of 85.7%. This suggests that higher temperatures improve the reduction process, likely promoting more efficient interaction between carbon and the iron ore.

However, increasing the temperature further to 900 °C caused a drop in recovery to 68.6%, even though there was a decline in grade to 64.8% Fe. This reduction in recovery indicates that excessively high temperatures might result in the re-oxidation of reduced iron or the creation of undesirable phases, potentially undermining overall efficiency.

These results underscore the significance of optimizing roasting temperature. The ideal temperature seems to be approximately 800 °C, where both grade and recovery reach their highest levels. Therefore, precise temperature control is essential for maximizing efficiency in the reduction roasting of iron ore, weighing the advantages of elevated temperatures against possible disadvantages.

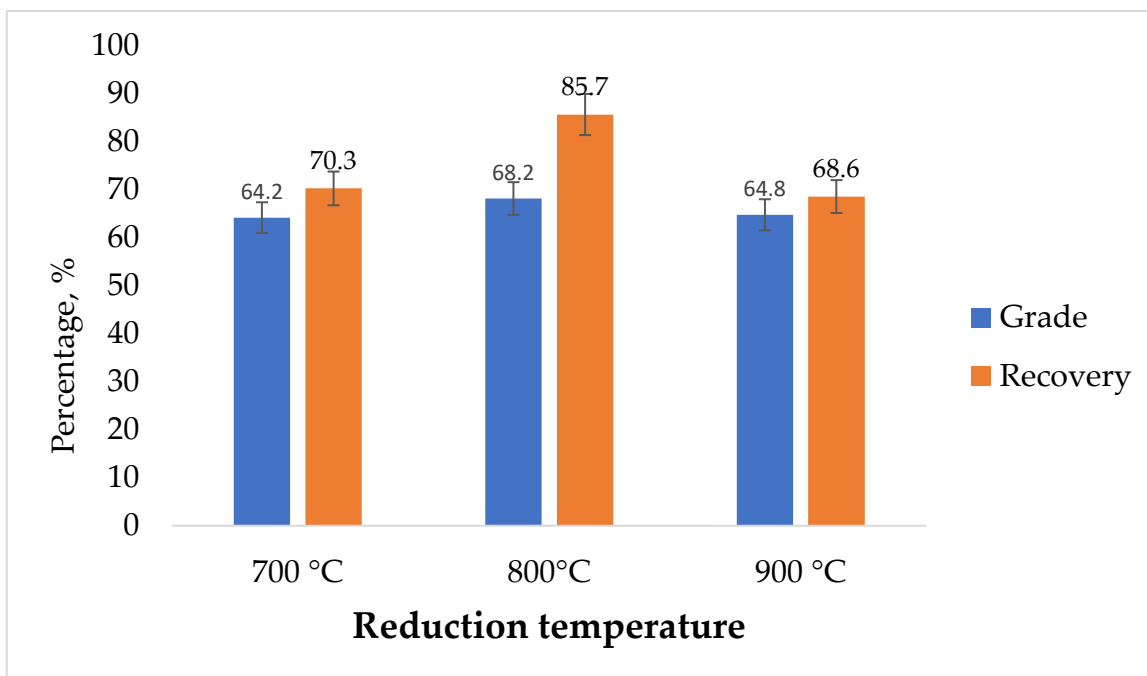


*Figure 26. Roasting temperature effects on reduction roasting.*

Higher temperatures facilitate reduction reactions due to their endothermic nature. Elevated temperatures lead to increased equilibrium constants and improve the conversion of iron oxides into metallic iron.

### **Effect of residence time**

In examining the impact of residence time during the reduction roasting of iron ore, changes in time significantly affect both the grade and recovery of iron, as illustrated in the figure. At a residence time of 30 minutes, the grade reached 64.1% Fe, with a recovery of 73.2%. Extending the residence time to 60 minutes led to a significant improvement in both metrics, resulting in a grade of 68.2% Fe and a remarkable recovery rate of 85.7%. This suggests that a longer residence time facilitates more complete reduction of the ore, thereby enhancing the extraction process. Fig. 27 demonstrates the correlation between grade & recovery vs residence time.

However, increasing the residence time to 90 minutes resulted in a decrease in recovery to 64.8%, even though a significant grade of 66.8% Fe was maintained. This reduction in recovery indicates that prolonged residence time may cause the re-oxidation of reduced iron or the generation of unwanted by-products, negatively impacting the overall efficiency of the process.

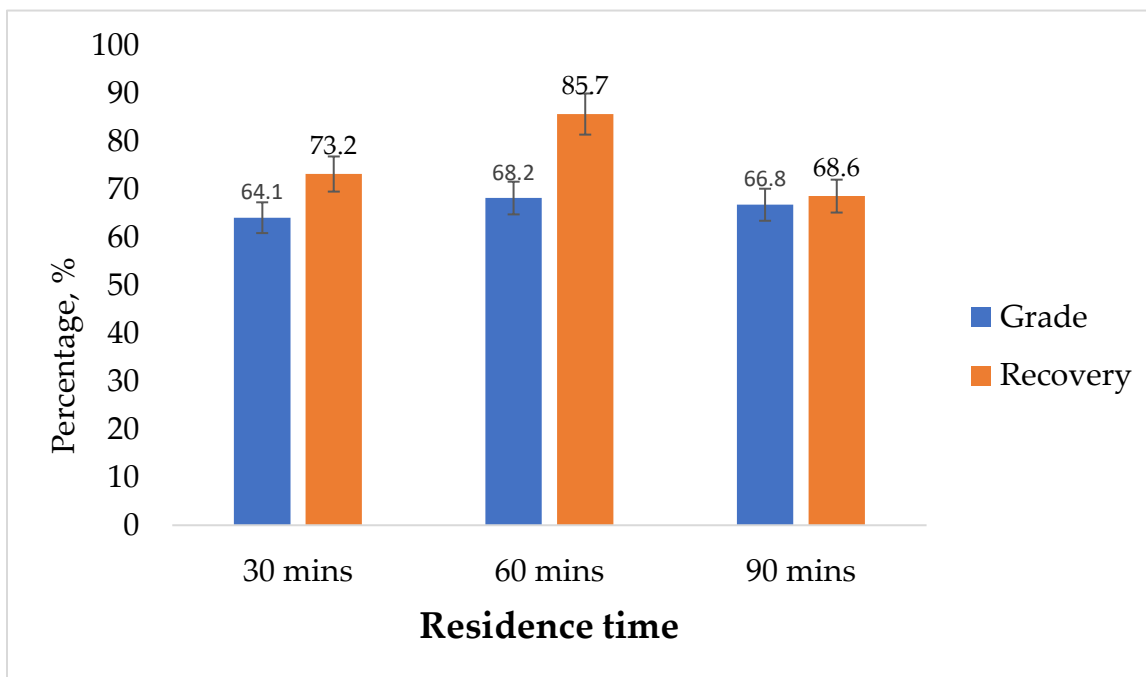


*Figure 27. Residence time effects on reduction roasting*

Thus, although an increase in residence time initially improves both grade and recovery, the findings highlight the importance of optimizing this parameter to prevent diminishing returns. The ideal residence time seems to be approximately 60 minutes, where both grade and recovery peak. This conclusion underscores the necessity for precise control of roasting conditions to achieve optimal results in iron ore reduction processes.

### **Effect of coal dosage**

The impact of coal dosage during the reduction roasting of iron ore, followed by low-intensity magnetic separation, is illustrated in Fig. 28. Both the Fe grade (%) and recovery (%) of reduced iron ore increase in a relatively linear manner as the reductant ratio rises, indicating a higher fixed carbon/oxygen ratio. At the optimal temperature of 800°C and an optimal residence time of 60

minutes, the iron grade significantly increased with higher coal dosages. The highest Fe grade of 68.2% was achieved with the maximum coal dosage of 0.5%, while the lowest dosage of 0.25% resulted in a grade of 65.7%. Additionally, the highest recovery of 85.7% for the reduced samples was also attained with the maximum coal dosage.

Increasing the FC/O ratio enhances the reduction of iron oxides to metallic iron, facilitating the effective removal of non-metallic products through magnetic separation. Higher coal dosage provides extra carbon for the reduction reactions. However, excessive coal can lead to incomplete combustion and the formation of unwanted by-products.

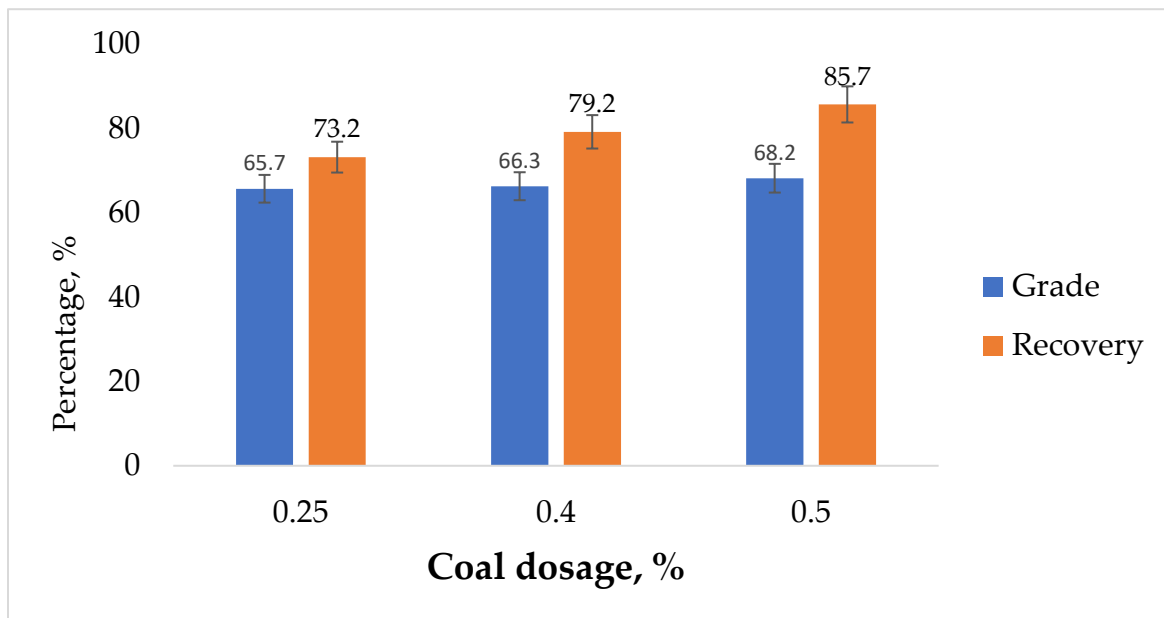


Figure 28. Coal dosage effects on reduction roasting of iron ore

#### 4.6 Statistical modelling

Following the roasting and magnetic separation processes of the samples, response variables of this study were determined as iron grade, which was studied using the XRF technique, with the iron grade which was calculated using equation (15).

The marginal means of S/N ratios were graphed for each factor across different levels, as shown in Fig. 29. The figure additionally shows the delta values (the difference between the highest and lowest ratios) for each factor, along with their assigned ranks.

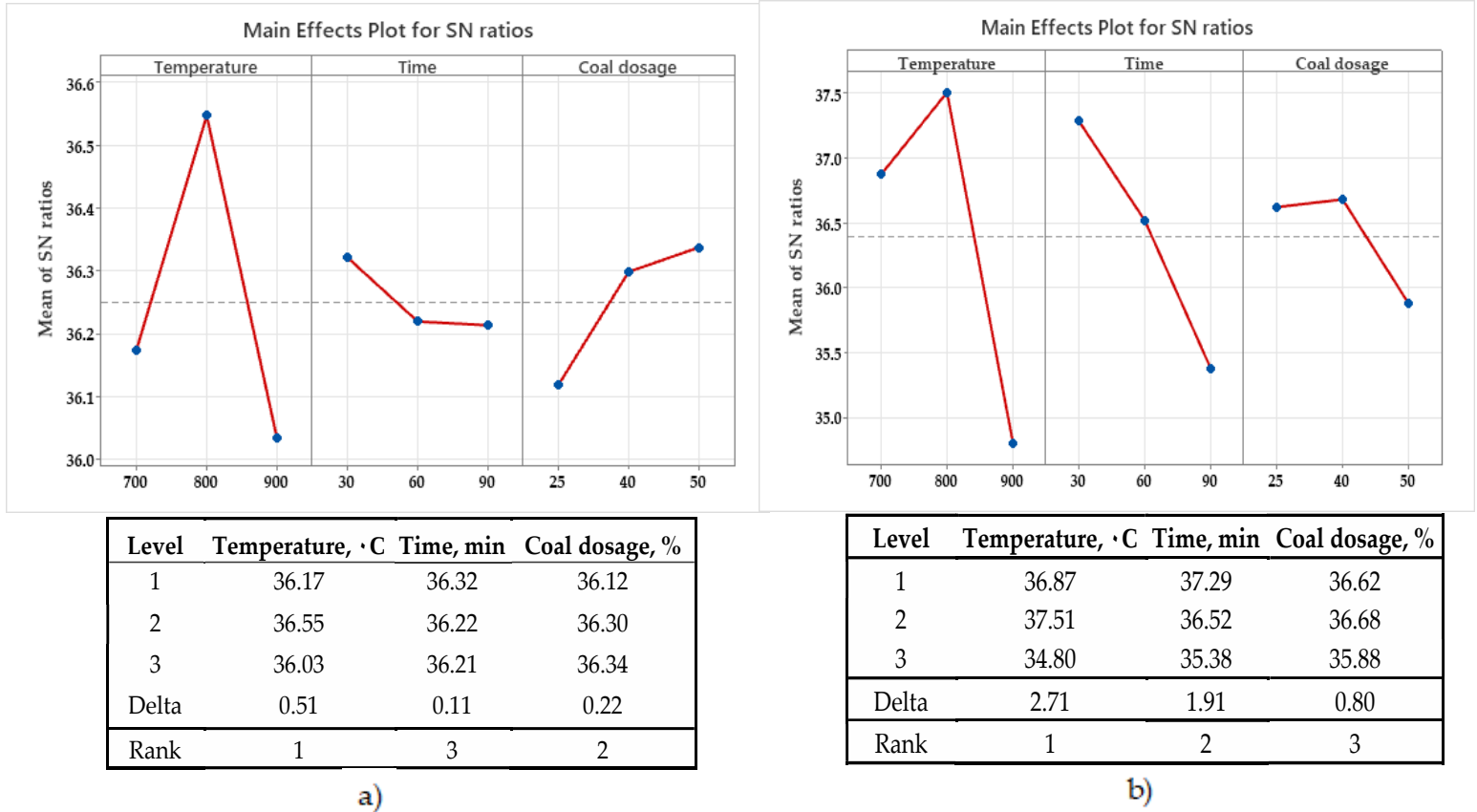


Figure 29. Taguchi main effects plot showing the S/N ratios for various factors in the muffle furnace roasting experiments: a) grade response and b) recovery response.

These ranks indicate the relative significance of the factors in the process. It is evident that the response ratio significantly increases with rising roasting temperature up to two levels, and with the reductant-to-feed ratio up to three levels. In contrast, from level 1 to 3, there is a minor, insignificant decrease in response as residence time increases. Temperature, with the highest delta value of 0.51, has the most substantial effect, followed by the reductant-to-feed ratio (delta value of 0.22) and time (delta value of 0.11).

Fig. 29(a) shows that the optimum experimental conditions for obtaining the highest Fe grade according to the Taguchi DoE are roasting temperature of 800 ° C, coal dosage: 50%, and roasting for 30 minutes. However, the highest recovery is represented by a temperature of 800 ° C, coal dosage: 40% and reduction time: 30 min (Fig. 29(b)). Taguchi also predicted that with these optimum combinations 68.4% Fe grade will be obtained at ~85% recovery.

#### 4.7 Thermodynamic and reaction kinetics modelling and experimental validation

##### 4.7.1 Kinetic Models & Model Fitting

The findings, presented in Fig. 30, indicate that the chemical reaction control model best represents the reduction process (Fig. 30a). As shown in Table 15, the regression coefficient ( $R^2$ ) for the chemical reaction control model at 800°C was 0.986, significantly higher than the ( $R^2$ ) for the product layer diffusion control model (0.975). This strong linear fit suggests that the chemical reaction at the surface of the iron oxide particles is the rate-controlling step, rather than diffusion through the product layer.

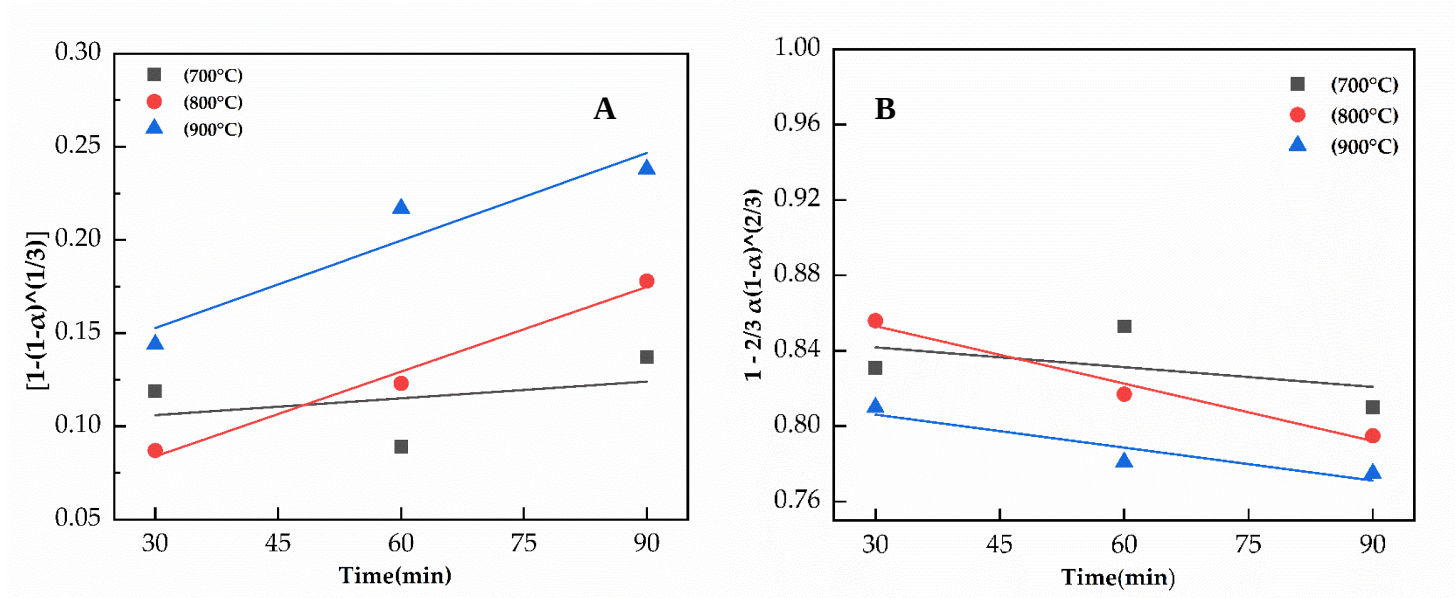


Figure 30. Plots of **a)**  $1 - (1 - \alpha)^{1/3}$  and **b)**  $1 - \frac{2}{3} \alpha(1 - \alpha)^{2/3}$  vs time for different temperatures.

Table 15. Regression coefficients and slope values of graphs in Fig. 30a and 30b

| Temperature | Slope $(1 - (1 - \alpha)^{\frac{1}{3}})$ | Regression coefficient ( $R^2$ ) | Slope $(1 - \frac{2}{3}\alpha(1 - \alpha)^{\frac{2}{3}})$ | Regression coefficient ( $R^2$ ) |
|-------------|------------------------------------------|----------------------------------|-----------------------------------------------------------|----------------------------------|
| 700°C       | $0.0003 \pm 7.50555\text{E} - 4$         | 0.138                            | $-0.00035 \pm 6.25463\text{E} - 4$                        | 0.238                            |
| 800°C       | $0.00152 \pm 1.82828\text{E}-4$          | 0.986                            | $-0.00102 \pm 1.63583\text{E} - 4$                        | 0.975                            |
| 900°C       | $0.00157 \pm 5.0037\text{E}-4$           | 0.907                            | $-5.83333\text{E}-4 \pm 2.21318\text{E}-4$                | 0.874                            |

The slopes of the linear plots from the chemical reaction control model (Table 15) further support this conclusion. The higher slope values at elevated temperatures (800°C and 900°C) indicate that the chemical reactions accelerate with increasing temperature, which is consistent with the Arrhenius equation and the general behaviour of thermally driven reactions.

(Muriana, Muzenda and Abubakre, 2014; Mishra, 2020) examined the reduction kinetics of iron ore using non-coking coal and found that the chemical reaction control model was the predominant mechanism at temperatures above 800°C, similarly to this study. Both studies highlight the role of surface reactions as the primary rate-controlling step in the reduction of iron oxides, reinforcing the conclusions drawn from this work.

From the data in Table 16, the rate constants ( $k$ ) increased with temperature, illustrating the temperature dependence of the reduction process. For example, at 1073.15 K (800°C), the rate constant after 60 minutes was  $0.00152 \text{ min}^{-1}$ , whereas at 1173.15 K (900°C), the rate constant for the same time was  $0.00157 \text{ min}^{-1}$ . This trend aligns with the Arrhenius behavior, where reaction rates increase exponentially with temperature due to more frequent and energetic molecular collisions.

Table 16. Rate constants

| Temperature, (k) | Time, (min) | Rate Constant (k)                     | ln(k) |
|------------------|-------------|---------------------------------------|-------|
| 973.15           | 30          | $1.5\text{E-}4 \pm 3.75278\text{E-}4$ | -8.80 |
| 973.15           | 60          | $3\text{E-}4 \pm 7.50555\text{E-}4$   | -8.11 |
| 973.15           | 90          | $4.5\text{E-}4 \pm 1.12583\text{E-}3$ | -7.70 |
| 1073.15          | 30          | $0.00076 \pm 9.14141\text{E-}5$       | -6.88 |
| 1073.15          | 60          | $0.00152 \pm 1.82828\text{E-}4$       | -6.49 |
| 1073.15          | 90          | $0.00228 \pm 2.74242\text{E-}4$       | -6.18 |
| 1173.15          | 30          | $0.00078 \pm 2.50185\text{E-}4$       | -6.85 |
| 1173.15          | 60          | $0.00157 \pm 5.0037\text{E-}4$        | -6.46 |
| 1173.15          | 90          | $0.00236 \pm 7.50555\text{E-}4$       | -6.15 |

Table 17 presents chemical controlled model activation energies ( $E_a$ ) for temperatures of 973.15-1173.15 K, calculated as 159.1 kJ/mol (90 minutes), 160.3 kJ/mol (60 minutes), 160.6 kJ/mol. (30 minutes) falls within the typical range for metallurgical reactions (20–100 kJ/mol). The activation energies are quite similar across the three residence times with a minor decrease in activation energy with increasing residence time suggests that the energy barrier slightly reduces at longer residence times. These values (ranging from 159.1 kJ/mol to 160.6 kJ/mol) are consistent with the expectations for solid-state reduction reactions in the ironmaking process, where activation energies typically range between 150-250 kJ/mol, depending on factors such as ore type, particle size, and reducing agent.

A study on iron ore reduction using coal found activation energies in the range of 140-180 kJ/mol for similar gas-solid reduction reactions involving iron oxides and carbon-based reductants(including coal) which aligns closely with the findings of this study (Hammam *et al.*, 2021). In reduction experiments on hematite using coal reported by (Liu *et al.*, 2014) activation

energies were also observed between 160-190 kJ/mol which is consistent with these results indicating a similar kinetic pathways.

As illustrated by the plot (Fig. 31; Fig 32; Fig. 33), the relationship between  $\ln(K)$  and  $1/T$  is linear, allowing for the calculation of activation energies across different residence times

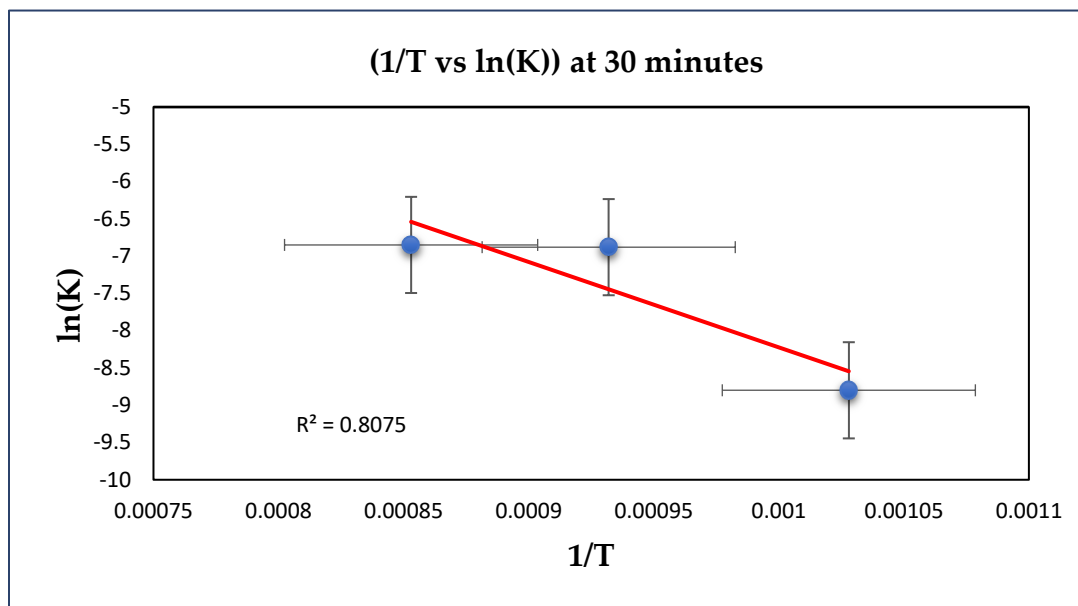


Figure 31.  $1/T$  vs  $\ln(K)$  plot for the chemical reaction control model (30 minutes)

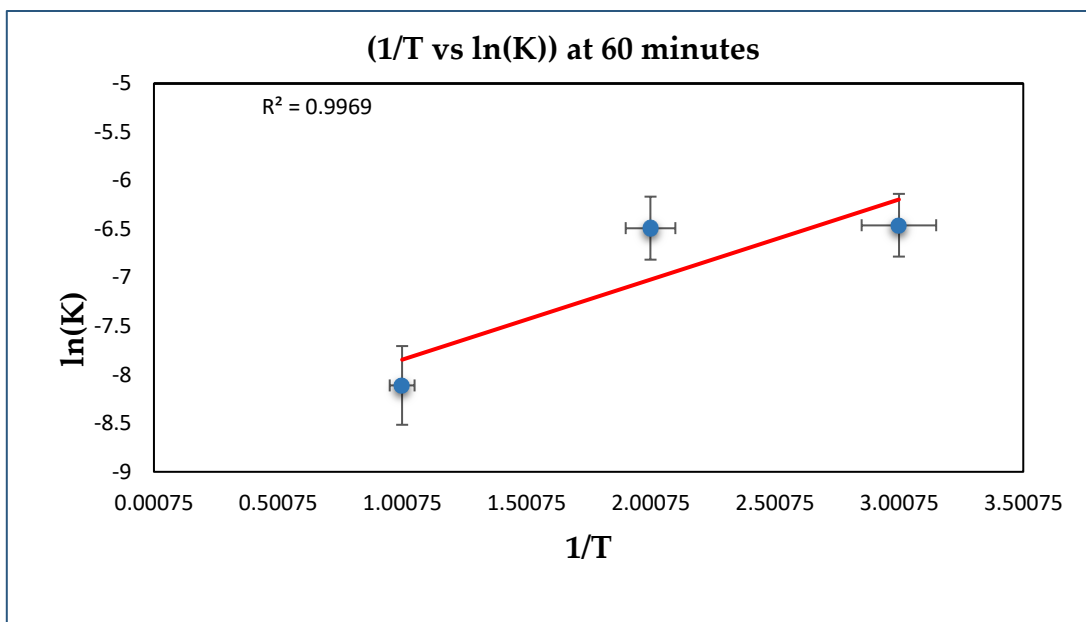


Figure 32.  $1/T$  vs  $\ln(K)$  plot for the chemical reaction control model (60 minutes)

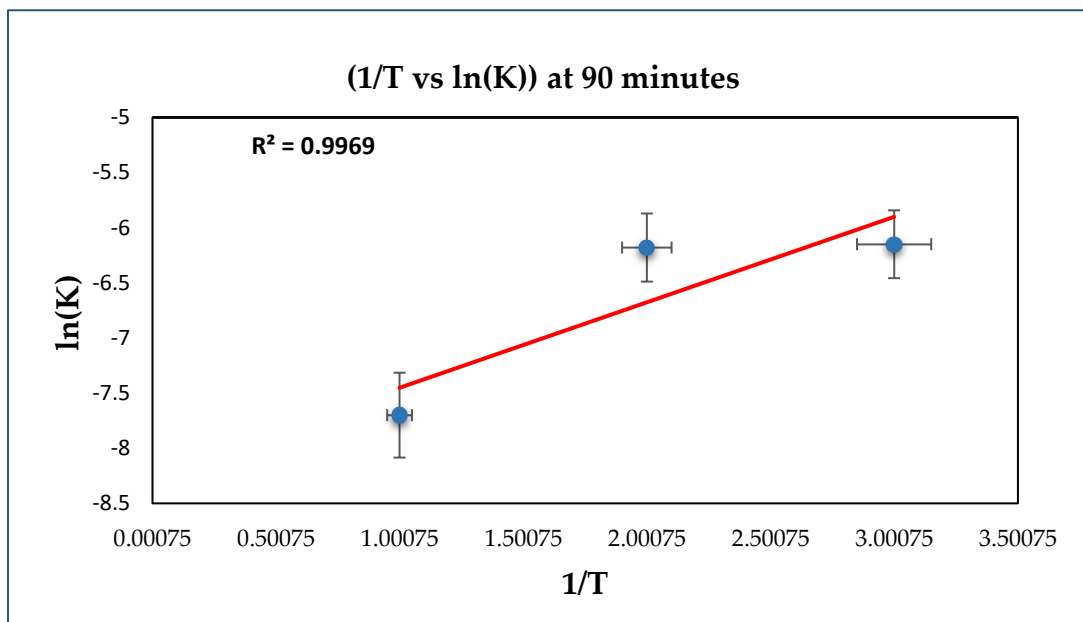


Figure 33.  $1/T$  vs  $\ln(K)$  plot for the chemical reaction control model (90 minutes)

Table 17. Chemical reaction control model activation energies (700, 800, 900 °C).

| Time    | <i>Ea(kj/mol)</i> | Slope   |
|---------|-------------------|---------|
| 30 min. | 160.6             | 19311.4 |
| 60 min. | 159.1             | 19134.7 |
| 90 min. | 160.3             | 19282.0 |

#### 4.7.2 Thermodynamics

Using the standard thermodynamic data, the values of  $\Delta G$  and  $\Delta S$  are obtained as:

$$(\Delta H) = -25.1 \text{ kJ/mol} = -25100 \text{ J/mol (conversion to J)}$$

$$(\Delta S) = -13.1 \text{ J/mol} \cdot \text{K}$$

#### Calculations:

$$\text{Example: } \Delta G(1073.15\text{K}) =$$

$$\Delta G = \Delta H - T\Delta S$$

$$\Delta G_{800} = -25100 \text{ J/mol} - (1073.15\text{K} * -13.1 \text{ J/mol} \cdot \text{K})$$

$$\Delta G_{800} = -25100 \text{ J/mol} + 14067.765 \text{ J/mol}$$

$$\Delta G_{800} = -11042.7 \text{ J/mol}$$

The equilibrium constant (K) for the reaction can be calculated using the equation:

$$\Delta G = -RT \ln K ; R \text{ is the universal gas constant (8.314 J/mol} \cdot \text{K)}$$

$$\ln K = -\frac{\Delta G}{RT}$$

$$\ln K = - \frac{-11042.7 \text{ J/mol}}{\left(8.314 \frac{\text{J}}{\text{mol} * \text{K}}\right) * (1073.15 \text{ K})}$$

$$K_{800} = e^{1.217} \approx \mathbf{3.37}$$

Rate Constant (k):  $k = Ae^{\frac{Ea}{RT}}$

$$A = e^{-0.12} \approx \mathbf{0.886}$$

Table 18. Gibbs Free Energy ( $\Delta G$ ), Equilibrium Constants, and Pre-exponential Factors (A) for Different Residence Times at Various Temperatures

| Temperature (K) | $\Delta G(\frac{\text{J}}{\text{mol}})$ | Equilibrium Constant (K) | Time (min) | Pre-exponential Factor (A) |
|-----------------|-----------------------------------------|--------------------------|------------|----------------------------|
| 973.15          | -12 351.74                              | 4.76                     | 30         | $2.17 \times 10^9$         |
|                 |                                         |                          | 60         | $1.89 \times 10^9$         |
|                 |                                         |                          | 90         | $2.12 \times 10^9$         |
| 1073.15         | -11 041.74                              | 3.37                     | 30         | $2.13 \times 10^8$         |
|                 |                                         |                          | 60         | $2.04 \times 10^8$         |
|                 |                                         |                          | 90         | $2.09 \times 10^8$         |
| 1173.15         | -9 731.74                               | 2.70                     | 30         | $5.40 \times 10^7$         |
|                 |                                         |                          | 60         | $5.02 \times 10^7$         |
|                 |                                         |                          | 90         | $5.29 \times 10^7$         |

The thermodynamic analysis of the reduction roasting process of iron ore using powdered non-coking coal reveals several important insights. The calculated Gibbs free energies ( $\Delta G$ ) of  $-12.35 \text{ kJ/mol}$  (973.15 K),  $-11.04 \text{ kJ/mol}$  (1073.15 K),  $-9.73 \text{ kJ/mol}$  (1173.15 K) indicates that the reaction is spontaneous and thermodynamically favourable under the specified temperature

and pressure conditions, suggesting a high likelihood of successful reduction since  $\Delta G < 0$ . This negative  $\Delta G$  suggests that the reaction will naturally proceed toward the formation of magnetite from hematite. The spontaneity of this reaction supports the primary objective of transforming hematite to magnetite, which is crucial for optimizing the process.

The equilibrium constants ( $K$ ) (4.76, 3.37, and 2.70) indicate that at all three temperatures, the products are favoured over the reactants, but the extent of product formation decreases as temperature increases. At 700°C,  $K = 4.76$  indicates a higher conversion of reactants to products compared to the higher temperatures. This suggests that lower temperatures are more thermodynamically favorable for the reaction.

At 900°C,  $K = 2.70$ , indicates the reaction still proceeds but with a lower extent of product formation than at 700°C. While increasing temperature generally increases the reaction rate (by lowering the activation energy barrier), the lower equilibrium constant at higher temperatures indicates a balance between kinetic and thermodynamic favourability.

The pre-exponential factor ( $A$ ) decreases as temperature increases for all residence times. This trend reflects that as the temperature rises, fewer high-energy collisions are required to surpass the activation energy barrier. This suggests that the reaction mechanism is efficient, and the reactants are likely to interact in a manner conducive to the successful reduction of hematite to magnetite. The combination of a moderate activation energy and a reasonable pre-exponential factor indicates that the process can be scaled effectively for industrial applications without the need for excessively high temperatures or reaction times.

The findings from this analysis are consistent with the existing literature on reduction roasting using carbonaceous materials as reductants. Previous studies have demonstrated that the use of non-coking coal or other carbon sources can effectively reduce iron ore to metallic iron due to the favourable thermodynamics of carbon reduction reactions (Sahu, Meikap and Biswal, 2022). Studies by (Faris *et al.*, 2017; Sahu *et al.*, 2019) report similar thermodynamic trends but with slight deviations in activation energy due to variations in ore reactivity and coal properties. These

studies also highlight the importance of coal properties, noting that non-coking coal often leads to more controlled reduction reactions compared to coking coal, which can exhibit higher rates of carbon monoxide generation.

## CHAPTER 5

### 5.1 Conclusions

This study focused on the beneficiation of low-grade iron ore through reduction roasting using non-coking coal as a reductant, subsequent low intensity magnetic separation and the following conclusions were drawn.

1. Mineralogical and characterization studies on the locally (Botswana) available low-grade iron ore and non-coking coal were conducted and further evaluated their suitability for the reduction roasting.
2. The reduction roasting process, utilizing locally sourced non-coking coal as the reductant, was employed to upgrade the low-grade ore iron (Fe) content up to 68% from 56% and these results were confirmed through Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), X-ray diffraction (XRD) and XRF
3. The key process parameters optimized were roasting temperature, residence time, and coal dosage based on the Taguchi method and the optimum values were determined through lab experiments to be **temperature: 800°C, residence time: 60 minutes** and **0.5% coal dosage**.
4. The activation energies ranging 159.1-160.6 kJ/mol were calculated for the reduction roasting of iron ore using coal at temperatures of 973.15 K to 1173.15 K and residence times of 30-90 minutes are consistent with values reported in the literature.
5. This study is unique to Botswana and provides a direction towards utilization of local mineral resources for long-term sustainability and economic development.

### 5.2 Future work scope

- Further research and pilot-scale trials are recommended to validate the scalability and techno-economic feasibility of the reduction roasting and magnetic separation process for industrial-scale application in Botswana.

- Investigate the potential for utilization of the non-magnetic tailings generated from the beneficiation process, such as in the construction or ceramic industries.
- Investigate the feasibility of utilizing alternative reductants, such as biomass or waste carbon sources, to enhance the environmental and economic sustainability of the beneficiation process.

### **5.3 Assumptions**

- The feed samples utilized in the experiments are representative of the broader local mineral resources.
- Laboratory conditions accurately reflect potential industrial processing environments.

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